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WHC-EP-0210 Rev 3

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Waste Characterization Plan for the Hanford Site Single-Shell Tanks

Appendix D - Quality Assurance Project Plan for Characterization of Single-Shell Tanks

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Ladies and Gentlemen:

REFERENCING WHC-EP-0210 REVS. 1, 2, AND 3

This memo is to inform you that when referencing the *Waste Characterization Plan for the Hanford Site Single-Shell Tanks*, (WCP) WHC-EP-0210 Revisions 1, 2, and 3, all three revisions should be included in the citation. This is to be done to avoid any further misunderstandings or miscommunications when citing the WCP. Because of the modular nature of the document, each revision is relatively self-contained; thus, the entire document has not gone through the revision/review cycle three times. The latter revisions rely on the previous editions for reference information and presume the reader's familiarity with the previous editions. Revision 1 consists of the primary text documenting the original purpose of the document and the procedures that were to be used as well as information that had yet to be determined. Revision 2 incorporated comments received on Revision 1 and also added *Appendix F: Sampling Test Plan for SSTs B-201, B-202 and T-203*; *Appendix G: Community Relations Plan for the Phase I-C Sampling of the Single-Shell Tanks*; and *Appendix H: Health and Safety Plan for Phase I-C Sampling of the Single-Shell Tanks*. Revision 3 was a self-contained addition of *Appendix I: Test Plan for Sampling and Analysis of Ten Single-Shell Tanks* and a limited update of *Appendix D: Quality Assurance/Quality Control Procedures*. Problems in citing the WCP will be corrected later this year when it undergoes a complete cover-to-cover revision.

Very truly yours,

H. D. Harmon, Vice President
Tank Waste Remediation System Division

pkc

RL - R. F. Christensen
J. M. Clark
R. D. Freeberg
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QUALITY ASSURANCE PROJECT PLAN FOR CHARACTERIZATION OF SINGLE-SHELL TANKS

APPENDIX D

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CONTENTS

CONTENTS (continued)

LIST OF FIGURES

LIST OF TABLES

LOCATOR PAGE

QAPjP Element	Supporting Document	Section(s)
Project Description	DOE/RL 89-16 WHC-EP-0210	1.0 and 2.0
Project Organization	WHC-EP-0294 QAPjP NO. SA-001 (PNL) WHC-SD-CP-QAPP-002	4.0 4.0 2.0
QA Objectives	QAPP No.SA-001 WHC-SD-CP-QAPP-002	5.0 3.0
Sampling Procedures	WHC-EP-0210 (Rev 2)	3.0 3.1 3.2
Sample Custody	QAPjP No. SA-001 (PNL) WHC-SD-CP-QAPP-002	5.0 7.0
Calibration Procedures	QAPjP No. SA-001 (PNL) WHC-SD-CP-QAPP-002	8.0 6.0
Analytical Procedures	QAPjP No. SA-001 (PNL) WHC-SD-CP-QAPP-002 WHC-EP-0210	9.0 7.0 5.0
Data Reduction Validation and Reporting	QAPjP No. SA-001 (PNL) WHC-SD-CP-QAPP-002 WHC-CM-5-3	10.0 8.0 2.0 2.2
Internal Quality Control Checks	QAPjP No. SA-001 (PNL) WHC-SD-CP-QAPP-002	11.0 9.0
Performance and System Audits	QAPjP No. SA-001 (PNL) WHC-SD-CP-QAPP-002	12.0 10.0
Preventive Maintenance	QAPjP No. SA-001 (PNL) WHC-SD-CP-QAPP-002	13.0 11.0
Data Assessment Procedures	QAPjP No. SA-001 (PNL) WHC-SD-CP-QAPP-002	14.0 12.0
Corrective Action	QAPjP No. SA-001 (PNL) WHC-SD-CP-QAPP-002	15.0 13.0
Quality Assurance Reports	QAPjP No. SA-001 (PNL) WHC-SD-CP-QAPP-002	16.0 14.0

2.0 PROJECT DESCRIPTION

The overall project objectives are described in the Draft Single-Shell Tanks System Closure/Corrective Action Work Plan (DOE/RL 89-16). The SST waste characterization project is responsible for sampling and analyzing the waste in Hanford Site's 149 SSTs to support regulatory, safety (waste reactivity) evaluation, performance assessment, waste retrieval and treatment technology development, supplemental environmental impact statement (EIS), and closure plan activities. The purpose of Phase I of the waste characterization project is to obtain information as quickly as possible on all 149 tanks to support the planning and development of the above activities. If this information is insufficient to support an activity, then additional waste characterization will be performed in Phase II with a revised QAPjP to meet any new objectives. The project addresses only characterization of the waste in the tanks and not the soils or the ancillary equipment associated with the tank system. Data from this program will be used to perform risk assessments and to evaluate disposal alternatives to support preparation of a supplemental environmental impact statement for the final EIS for the disposal of the Hanford Site defense high-level transuranic waste (DOE 1988). Initial data will be used to help develop DQOs to permit preliminary sorting decisions about those tanks wastes that may be treated in place and those that may be retrieved. These data permit the project to identify and evaluate additional characterization and waste treatment requirements. The project also will provide data to support regulatory and safety evaluations for storage and treatment of the waste.

The SST waste characterization project is only one part of a larger program to develop a system for final disposition of SST waste. Data from the SST characterization project will be used to support the development of final disposal alternatives. The development of these alternatives has been described in several system engineering documents (Aiken et al. 1990; Garfield[a] 1990; Garfield[b] 1990; Boomer et al. 1990) which also identify potential data users and their interactions. Further details on objectives for sampling and analyzing wastes in the tanks will be included in future test plans for characterizing the SSTs.

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3.0 PROJECT ORGANIZATION AND RESPONSIBILITY

Project organizations and responsibilities are described in the QA Program Plan for SST characterization (WHC 1990), the 222-S Laboratory QA project plan (WHC 1989), and the 325 Laboratory QA project plan (PNL 1989) for the project. The Westinghouse Hanford Company organizational structure involved in characterizing SST waste is outlined in the QA Program Plan (WHC 1990). However, the organizations described in these program plans and the responsibilities of these organizations are expected to change significantly in Fiscal Year 1992. These changes are currently pending approval from the U.S. Department of Energy and will be formalized after the issuance of this document. The sampling and analysis of SST waste samples are described with the following major operations:

1. Plan and request sampling.
2. Sample waste tanks.
3. Transport and receive sample.
4. Break down (extrude) and prepare (homogenize and composite) samples for analysis.
5. Analyze samples.
6. Verify and report results.
7. Validate data packages.

The Tank Waste Characterization Technology Section under Waste Management Technology is responsible for identifying the waste tanks to be sampled, for providing instructions to Tank Farm Plant Engineering for sampling the tanks; and to the Office of Sample Management for analyzing the samples. The Office of Sample Management (OSM) decides which Laboratory (222-S or 325) will do the work and provides instruction to the laboratory through a statement of work request.

Samples are taken by the Tank Farm Surveillance Operations group and transported to the designated laboratory. The Tank Farm Surveillance Operations group initiates the chain-of-custody procedure and prepares the field blanks. Samples are received in either the Westinghouse Hanford 222-S Laboratory or the Pacific Northwest Laboratory (PNL) 325 Laboratory where the sample custodian completes the chain-of-custody process and logs the sample into the laboratory. Subsampling of extruded core segments is performed by the Process Development Support Unit of the 222-S Laboratory and the Analytical Laboratory Operations group at PNL. These groups are responsible for logging in the samples and preparing the instructions (travelers) for sample breakdown and analysis.

Samples are broken down and extruded by the hot cell operators in the Process Chemistry Laboratory group for Westinghouse Hanford and the A Hot Cell

operators for the 325 Laboratory. These hot cell groups perform visual and physical measurements on the extruded samples, homogenize the samples, and prepare subsamples to be dispensed to the analytical chemistry groups for analysis. The subsamples for inorganic and radiochemical analyses are given to the 222-S Process Development Support Unit for analysis. Additional subsamples are provided to other members of the Process Chemistry Laboratory group for physical analyses such as differential scanning calorimetry (DSC) and particle size. Subsamples for organic and rheology analysis from 222-S Laboratory tank samples are sent to PNL for analysis since the 222-S Laboratory does not have this capability at this time. These capabilities are planned to be added in FY 1991.

Subsamples from the A Hot Cells at 325 Laboratory are provided to specific analytical teams at PNL for analysis. Samples from the A Hot Cell in 325 Laboratory are transferred to the B Hot Cell for analytical preparation procedures.

Each analytical team (inorganic, radiochemical, organic) is responsible for analyzing the samples and verifying the data quality in accordance with statement of work and the QC criteria for the project described in this QAPjP.

Data packages for segment and core composite analyses are prepared by the analytical groups of both laboratories and submitted to the Office of Sample Management for data validation before release to the data users.

4.0 QA OBJECTIVES FOR MEASUREMENT DATA

Data obtained in the waste characterization project will be used to support many activities and users. Some of these are identified below:

User	Activity
Westinghouse Hanford	
• Regulatory Analysis	Preliminary regulatory evaluation
• RCRA Permitting/NEPA Group	Supplemental environmental impact statement, and closure/post closure plans
• Waste Management Technology	Evaluation of disposal alternatives, treatment technology development, preliminary sorting of tanks, characterization planning
• Environmental Technology	Development of performance and risk assessment
• Systems Engineering	Design of waste retrieval equipment
• Waste Tank Safety, Operations and Remediations	Evaluation of tank safety conditions
Pacific Northwest Laboratory	
• Waste Technology Center	Support studies on above activities
Washington State and Department of Ecology	Oversight and evaluation of data decisions with respect to Washington Administrative Codes.
U.S. Department of Energy	Oversite and evaluation of data and decisions with respect to state and federal requirements

Each of these primary users may have additional secondary users with different data requirements such as the different technologies being studied for treatment and retrieval. It is the responsibility of these users to develop data quality objectives for the data used from the waste characterization program to ensure that the required information with the desired accuracy is obtained to solve their problem. It is the responsibility of the waste characterization team in Waste Management Technology to collect

these data objectives from the users and optimize a sampling and analysis design for characterizing the waste to meet as many of the data users needs as possible within the constraints of the sampling and analysis capabilities. Section 4.0 of the Waste Characterization Plan (WCP) (WHC-EP-0210) discusses the various parameters for analysis and the rationale for their selection. Potential uses of the data are summarized in Table 4-14 of the WCP based on previous knowledge of regulations or existing treatment processes.

Table D-1 provides a guideline for test being performed and the user of the data. In some cases the users have not specified their data requirements (none identified). These areas are expected to become better defined as these programs progress.

Inventory measurements of inorganic chemicals and radiochemicals receives the most wide-spread use. Even though pre-treatment users have not specified their requirements, it is anticipated they will be interested in analytes that produce most of the risk or analytes that may impact the performance of the treatment process. The inventory estimates are of interest to Ecology to evaluate waste designation using the toxic equivalent concentration calculation (TEC) which is a weighted sum of waste constituents based on their toxicity. This is the primary reason for performing regulatory organic analyses. Some of the analyte results obtained in inventory measurements will be used to assess the potential reactivity of the waste. Even though no specific analytes have been identified it is anticipated that oxidants and reduced carbon will be of interest.

Physical property measurements are primarily of interest to the groups developing retrieval technology. Waste hardness and viscosity are important in designing mechanical systems to remove the waste. The presence of drainable liquid is of regulatory interest.

Waste designation tests are primarily performed to satisfy regulatory requirements. The reactivity of the waste is a requirement of regulatory waste designation procedures and to tank farm operations for assessment of safety concerns in storing and sampling waste.

The determination of solubility and adsorption coefficient constants for the waste is primarily important to long term release risk performance assessment studies. These studies will impact characterization planning and tank sorting decisions. The information also will be used to evaluate the no action alternative for the supplemental environmental impact statement for the SSTs.

The sampling and performing of tests in duplicates is important to establishing the uncertainty in the test results. This uncertainty needs to be known to evaluate the significance of results and the accuracy of decisions. This information should be important to most users.

As described earlier one of the primary purposes of Phase I characterization was to perform a preliminary sorting or ranking of the tanks into those whose waste should be retrieved and those whose waste may be treated in place. Since this problem requires the use of both performance

Testing Objectives	Characterization Planning (Sorting)	Performance Assessment (EIS)	Retrieval	Pre-treatment	Regulatory	Safety
Inventory Tests Inorganic Organic Radiochemical	Analytes for LTRR, STIR and CLASS	Analytes for LTRR, STIR	None Identified	High risk analytes and process sensitive analytes (None Identified)	Waste Designation (TEC) metals, anions organics	Total Carbon Nitrate Ammonia Cyanide Water (None Identified) Ferrocyanide Speciation Chemical Oxygen Demand
Physical Properties Tests	None Identified	None Identified	Hardness Rheology Density Particle Size	None Identified	Drainable Liquid	Thermal Output Specific Heat Coefficient of Thermal Expansion XRD, PLM
Waste Designation Tests	None Identified	None Identified	None Identified	None Identified	TCLP Corrosion by (pH)	Reactivity by DSC
Transport Constant Test	Solubility Adsorbtion Coefficient	Solubility Adsorbtion Coefficient	None Identified	None Identified	None Identified	Thermal Conductivity of Sludge Thermal Conductivity of Frozen Sludge
Analysis of Errors	Decision Accuracy	Source Term Uncertainty	Decision Accuracy	Decision Accuracy	Decision Accuracy	Decision Accuracy
DSC = Differential Scanning Calorimetry; XRD = X-ray Diffraction; PLM = Polarized Light Microscope						

Table D-1. Matrix of Testing Objectives and Data Users.

assessment and regulatory requirements in the decision making process, it is expected to require the most comprehensive and restrictive data requirements. The data quality objectives described in this appendix will be the objectives needed to meet the most demanding user requirement. The process for arriving at the data quality objectives for the tank sorting problem are described below.

Data quality objectives for preliminary sorting or ranking of tanks for treat-in-place and retrieve decisions are being developed using information from a computer model (TRAC) of tank contents (Morgan 1988) and initial information from Phase IA and IB. The analyte priorities report (Wegeng 1990) describes how data quality objectives are being developed for the project. The DQO process has been described in several reports (EPA 1987, Neptune 1990[a], Neptune 1990[b]). The DQO logic process is provided below and the general DQO process for SST characterization is outlined in Table D-2. This table addresses the questions recommended by EPA for the development of DQOs. In most instances, reference is made to sections of this document or other documents where response to the question is addressed. Titles corresponding to the document numbers are stated in the legend at the end of the table.

DQO Logic Process

1. State the problem.
2. Identify a decision that addresses problem.
3. Identify inputs affecting decision.
4. Specify domain of decision.
5. Develop logic statement.
6. Establish constraints on uncertainty.
7. Optimize design for obtaining data.

The domain of the present project focuses on establishing the total inventory for chemical and radiochemical constituents in the tank. Some vertical sampling and analyses are planned to identify layering in the tanks for some analytical and physical parameters. The domain for this work is further defined in the scenario for release in the Multimedia Environmental Pollutant Assessment System (MEPAS) model which is an enhanced version of the Remedial Action Priority System (RAPS) (Droppo et al. 1989). The characterization problem being addressed is sorting the tanks into preliminary treat-in-place and retrieve candidates. Major factors affecting this decision have been identified as: long-term release risk (LTRR), short-term intruder risk (STIR), and waste classification (CLASS) based on both Nuclear Regulatory Commission (NRC) low level waste (LLW) classification limits, and on Ecology toxic equivalent concentration criteria (TEC).

The model RAPS/MEPAS (Droppo 1989) was used to identify the constituents most important from a long-term risk perspective. This preliminary screen used TRAC information (constituents and concentrations) as a basis for assessment. The model was also used to establish preliminary waste concentration thresholds (concentration threshold limits [CTL]). Concentration thresholds are defined as the concentration of a constituent in the waste that produces 1% of the total risk index. These CTLs help to define detection limit requirements for the project. Decision simulation

calculations are in progress that will help define the number of samples and duplicates needed to make confident decisions. These initial evaluations are based on TRAC estimates and unverified estimates of solubilities and soil adsorption coefficients (R_d) which need to be substantiated with evaluations using actual analyses of tank waste and verified solubility and R_d data. This work will lead to a logic statement and an optimized design for obtaining data.

The development of the DQOs is an interactive process and will improve as better and more representative estimates of tank inventories and performance assessment parameters are obtained. The DQOs for the project should be driven by the decision requirements and not by analytical capabilities.

Table D-3 identifies the constituents that are expected to contribute to the long-term (LTRR) and short-term risk (STIR) and classification (CLASS) criteria. Analytes that contribute between 1.0 and 99% of the cumulative risk index are classified as Type I. Analytes that contribute from 0.01 to 1.0% of the risk index are identified as Type II. Analytes that contribute to less than 0.01% of the cumulative risk index are Type III analytes. The procedures used to select these analytes are described in the analyte priorities report (Wegeng 1990). For comparison purposes, the analytical procedures for these parameters and their capabilities also are provided. In many cases TBD (to be determined) has been denoted for many values. These values will be identified after Phase IA and IB data have been completely analyzed and will be used as the baseline for analytical capability. Quality control information will be gathered during Phase IC and will make analytical capability estimates more representative for all the tanks.

Most of the metals in Table D-3 are performed by inductively coupled plasma optical emission spectroscopy (ICP) analysis; however, the table also includes other more sensitive atomic absorption (AA) and fluorometric methods following the ICP method. Even though Type II and Type III parameters may not be as important to the decision criteria as Type I, they are included when they are obtained automatically with multi-element techniques such as ICP. Other elements such as cobalt, copper, magnesium, manganese(VII), selenium, tin, thallium, and zinc, which were not considered or found important in the RAPS/MEPAS evaluation have been included because they are important to regulatory standards and can be estimated with ICP analysis. Thorium analysis is included because it can be determined by ICP easier than by radiochemical methods, and is used to verify that this is not a major component in the waste. The detection limits in Table D-3 are based on what can be measured in the sample, i.e. instrument detection limits multiplied by applicable dilution factors for the normal preparation method. However, they do not account for matrix interferences and backgrounds that may vary between samples. The detection limits in Table D-3 are based on preliminary assumptions for analytical procedures being used and may change when more accurate detection limit information is available. The concentrations threshold limits are those concentrations derived in the analyte priorities document (Wegeng 1990) based on quantities that would produce 1% of the cumulative risk index. Some of the concentration threshold values are based on regulatory limits (sulfide), on limits established in the WCP (NH_3, organics) and on specifications by the user (physical measurements). The analyte priority study (Wegeng 1990) recommends detection limit goals based on the CTLs and on the source (LTRR,

Table D-2. Summary of Data Quality Objectives (DQO).

DQO Questions	Responses/Reference Documents
1. STATE PROBLEM	Final disposition of the SST system.
1a) How was site used historically?	WHC-EP-0210 Section 2 DOE/RL-89-16 Sections 1 and 2 WHC-EP-0338 Section 1
1b) What were emergency remedial response or actions taken: • What actions • When • How accomplished?	DOE/RL-89-16 Section 7
1c) Based on current observations of site conditions, does site differ from conceptual models? If so, how has it changed?	Phase IA, IB sampling results, which are still under evaluation and future Phase I sampling results, will indicate if tank inventories differ from TRAC estimates. Reference: TBD.
1d) What are the known and suspected sources of contamination?	See response to 1a above. DOE/RL-89-16 Section 4 WHC-EP-0352 Section 2 WHC-EP-0338 Section 2
1e) What is the initial list of known and suspected site-related contaminants?	See response to 1d above.
1f) What concentrations of known and suspected contaminates have been measured at the site?	See response to 1d above. Actual sampling inside of tanks is part of Phase I and II waste characterization program.
1g) What are the available ARARs for the known and suspected contaminants?	WAC 173-303 WHC-EP-0338 Section 2 PNL-7426 Section 8 Above documents list currently known applicable requirements. Additional requirements may need to be developed in the future. There could also be mutually conflicting requirements which need to be resolved.
1h) By what routes might contaminate migrate offsite?	Tank Leaks. DOE/RL-89-16 Sections 2, 4, and 8

Table D-2. Summary of Data Quality Objectives (DQO).

DQO Questions	Responses/Reference Documents
1i) What population is potentially exposed to known and suspected site-related contaminates?	TBD.
1j) Based on available information, state the potentially significant reasonable maximum exposure pathway scenarios you would like to test.	TBD.
1k) Describe sources of information used to define problem.	TBD.
2. IDENTIFY A DECISION THAT ADDRESSES THE PROBLEM	In-situ disposal or retrieval alternatives.
2a) What are the alternative actions to address the potential problem?	DOE/RL-89-16 Section 10 WHC-EP-0338 Section 1
2b) What is the decision that will be the focus of this survey?	The results of the survey will not directly affect the decision between the above disposal alternatives. However, they will be used to develop methodologies for establishing tank inventories which is a prerequisite for decision making, irrespective of the alternative chosen.
3. IDENTIFY INPUTS AFFECTING THE DECISION	
3a) Is it safe to assume that environmental data is important for decision making or political/social considerations are also important?	The political and social consideration are factors in the TPA. The considerations affecting the disposal decisions are: long-term release risk (LTRR), short-term intruder risk (STIR), regulatory waste classification, and cost/schedule considerations. WHC-EP-0210 Appendix D, Section 4 PRR, PNL-7573 DOE/RL-89-16 Section 10

Table D-2. Summary of Data Quality Objectives (DQO).

DQO Questions	Responses/Reference Documents
3b) What questions about the environment must be answered to make the decision?	LTRR, STIR, and regulatory waste classification. See 3a above.
3c) Will action levels (decision criteria for determining if a problem exists) for contaminates be determined by risk-based calculations?	Yes. Performance assessments will be preformed. PRR, PNL-7573 WHC-EP-0338 Section 1, Figure 1-2.
4. SPECIFY DOMAIN OF THE DECISION	The domain is each individual tank.
4a) What are the receptor populations for which risk assessment will be calculated for preliminary action levels?	For the short term of the waste characterization program, it is the site workers. There are no trespassers because of the security. PRR, PNL-7573
4b) What are the current and future land uses?	Currently a Federal reservation; it will continue to be so for the foreseeable future.
4c) What are the anticipated or known receptor population activity patterns associated with current and future land use?	DOE/RL-89-16 Section 10.4
4d) What is the smallest area over which a receptor might limit its activities during the period over which exposure is possible?	Restricted access limited to site workers.
4e) If site consists of multiple exposure units, will decisions be made on discrete areas or entire site?	A decision will apply to each individual tank.
4f) Are you interested in a scale of resolution smaller than an exposure unit?	No. The entire tank is one uniform exposure unit.
4g) Does site historical information allow estimation of hot spots?	N/A. See response to 4f above.
4h) What is the minimum size of hot spot?	N/A. See response to 4f above.

Table D-2. Summary of Data Quality Objectives (DQO).

DQO Questions	Responses/Reference Documents
4i) When will data be collected and what timeframe will it represent?	WHC-EP-0210 Section 3
5. DEVELOP LOGIC STATEMENT	
5a) How will data collected at this tank be summarized for use in answering the main question?	PRR, PNL-7573
5b) If ARARs or RfDs are to be used, what are the thresholds of potential concern?	WHC-EP-0210, Appendix F PRR, PNL-7573 The sampling of initial tanks in Phase 1C is to determine the presence or absence of certain analytes of preestablished priorities. It is not intended to determine whether these analytes are above or below established regulatory threshold levels.
5c) If risk based criteria are used, the following questions must be answered (apportioning risk, target daily intakes, etc.).	Risk based criteria will be used for selecting final disposal options but not during waste characterizations.
5d) Clarify initial key questions incorporating the specific domain, decision criteria, and summary statistic.	Quantitative decision criteria will be established during Phase II sampling. Reference EP-0338 Section 1 for decision logic of disposal alternatives.
5e) Create logic statement(s) in if/then terms that explain what actions will be taken under what circumstances.	See response to 5d above.
6. ESTABLISH CONSTRAINTS ON UNCERTAINTY	
6a) What are the potential consequences of incorrectly deciding the site is not a problem (false negative).	N/A. See responses to 2b and 5b above. PRR, PNL-7573

Table D-2. Summary of Data Quality Objectives (DQO).

DQO Questions	Responses/Reference Documents
6b) What are the potential consequences of incorrectly deciding the site is a problem (false positive).	See response to 6a above.
6c) For each potential consequence, what is the qualitative discomfort of making the error?	See response to 6a above.
6d) What concentration of site-related contaminates of potential concern correspond to following increments of risk levels.	See response to 6a above.
6e) Translate qualitative levels to quantitative values--for false negative.	See response to 6a above.
6f) As above, for false positive.	See response to 6a above.
6g) Specify true values for which either decision error can be tolerated.	See response to 6a above.
7. OPTIMIZE DESIGN FOR OBTAINING DATA	
7a) What are the estimated variables and distribution of site-related contaminates of potential concern?	The purpose of the waste characterization is to answer this specific question. The sampling of tanks in Phase IC is part of the waste characterization effort.
7b) What is the lowest cost sampling plan to achieve desired constraints on uncertainty?	WHC-EP-0210 Sections 3, 4, and 5; Appendixes D and F PRR, PNL-7573

Table D-2. Summary of Data Quality Objectives (DQO).

DQO Questions	Responses/Reference Documents
LEGEND	
WHC-EP-0210	Waste characterization plan for the Hanford Site Single-Shell tanks (this document).
DOE/RL-89-16	(Draft) Single-Shell Tanks System Closure/Correction Action Work Plan.
WHC-EP-0338	Functional requirements baseline for the closure of Single-Shell Tank.
WHC-EP-0352	
WAC-173-303	Washington State Department of Ecology, Dangerous Waste Reg. "89."
PNL-7426	System analysis of alternative for final disposition of the Single-Shell Tank System on the Hanford Site.
PNL-7573	Preliminary Recommendations on the Design of the Characterization Program for the Hanford Site Single-Shell Tanks--A System Analysis.

STIR or CLASS) of the most restrictive CTL. This report recommends detection limits 100 times less than the CTL for LTRR sensitive analytes and 10 times less than the CTL for STIR and CLASS sensitive analytes. Table D-4 assesses the present estimated detection limits for each analyte against these criteria. Based on this evaluation improved detection limits for the following analytes may be needed: As, Al, Be, Bi, Cr, Pb, Sb, NO_3^-, NO_2^-, F^-, Cl^-, ^{14}C, ^{99}Tc, ^{129}I, ^{137}Cs and the actinides. Since Al, Bi, Cr, NO_3^-, NO_2^-, F^-, and ^{137}Cs will probably be present in many of the wastes at high levels, the detection limits for these analytes may not be an issue. The concentration threshold limits could change if the solubility and transport (R_d) properties of the elements are different from those used in the model. Further evaluation of the performance assessment model based on actual waste concentrations and transport properties may be required before the importance of improving the detection limits can be determined.

Most of the radionuclide detection limits are about equal to the concentration threshold limits using normal sample sizes and counting times. The methods for ^{129}I and ^{99}Tc may require significant improvements to meet CTLs. Further evaluation of the radiochemical methods and the CTLs are needed to identify development needs.

Most of the anion methods do not have detection limits 10 times below the CTLs except for sulfate; however, most of the anion detection limits are below or near the concentration threshold limits.

Other analytical methods that need to be developed are: ferrocyanide speciation, X-ray diffraction, polarized light microscopy, determining the coefficient of thermal expansion, specific heat, chemical oxygen demand,

thermal conductivity, and thermal conductivity of the frozen sludge. DQOs and detection limits for these procedures have yet to be determined.

Analytical methods for determining the concentration of complexants have not been developed. Additionally meeting CTLs based on total organic carbon (TOC) analyses and high performance liquid chromatography (HPLC) methods is expected to be inadequate because of the sensitivity or selectivity of the methods. Other analytical methods that need to be developed are: Ferrocyanide Speciation, Coefficent of Thermal Expansion, Specific Heat, Chemical Oxygen Demand, X-ray Diffraction, Polarized Light Microscopy, Thermal Conductivity, and Thermal Conductivity of frozen sludge. DQOs and detection limits have yet to be determined.

Data quality objective accuracy and precision requirements will be determined from decision simulation studies now in progress. Complete evaluation of these DQOs will not be available until all the Phase IA/IB data have been analyzed. Analytical capability information will be used until better guidance is available.

Since solid laboratory standards are not available, accuracy for solids will be monitored using spike recovery performance. Laboratory accuracy for liquid samples will be established from the analysis of laboratory control standards (LCSs). Values for these accuracies are initially based on Phase IA/IB results and updated with routine quality control results. Precisions for the laboratory capabilities will be based on 3σ limits for analysis of duplicate sample aliquots of solids and on repeated single analyses of LCSs for liquids.

Completeness is estimated to be 90% for all analyses. However, since Type II and III results are not as important as Type I in making decisions, completeness criteria for these parameters have been set to 75% and 50%, respectively, to indicate the level of importance of these parameters. This indicates that failure to achieve a 90% goal of valid data for these parameters does not require the same response as for Type I constituents. For example, occasionally one of the many channels on a direct reading ICP may fail to produce adequate data. If this channel is for a Type I parameter, analyses shall be suspended and the condition corrected to meet the 90% completeness goal. However, if the channel is a Type II or Type III parameter, suspending the analyses may not be required or even worthwhile.

All data shall be reported in μg/g or μCi/g of dry sample weight for all solids and μg/mL or μCi/mL for liquid samples. The weight percent water correction factor used to correct the data from wet weight to dry weight shall be included with each batch of data. Toxic characteristic leach procedure (TCLP) results shall be reported as mg/L of the extract.

Single-shell tanks will contain relatively high concentrations of water in the sludge or crystalline matrix. Because of the potential for water loss during handling in highly ventilated radiation facilities, the weight percent water and the preparation for the analysis shall be performed in the same time frame to provide the best comparability. If over 30 days elapses between determining the weight percent water and preparing an aliquot for analysis, the weight percent water shall be rerun providing adequate sample is

available. Results of this analysis would then be used to correct any later analyses to a dried weight basis. The laboratory shall make every effort to keep the time the sample and subsample aliquots are open to the atmosphere at a minimum and to keep them sealed to prevent water loss.

Table D-3. Data Quality Objectives and Laboratory Capabilities. (sheet 1 of 10)

Parameter	Preparation WHC PNL	Type/basis	Method WHC PNL	Threshold concentration µg/g	Detection limit µg/g	DQO accuracy	DQO precision	Laboratory accuracy solid liquid	Laboratory precision solid liquid	Completeness
Inorganic metals (µg/g or µg/mL)										
ICP As	LA-505-159(A) PNL-ALO-101(A)	II/LTRR	LA-505-151 PNL-SP-7	0.2	3.3 30	TBD	TBD	TBD TBD	TBD TBD	75%
HYAA As GFAA As	Direct PNL-ALO-101(A)	II/LTRR	LA-355-131 PNL-ALO-214	0.2	0.03 1	TBD	TBD	TBD TBD	TBD TBD	75%
ICP Ag	LA-505-159(A) PNL-ALO-101(A)	II/LTRR	LA-505-151 PNL-SP-7	186	2.7 2	TBD	TBD	TBD TBD	TBD TBD	75%
ICP Al	LA-505-159(A) PNL-ALO-101(A)	I/CLASS	LA-505-151 PNL-SP-7	63	11 10	TBD	TBD	TBD TBD	TBD TBD	90%
ICP Ba	LA-505-159(A) PNL-ALO-101(A)	I/STIR	LA-505-151 PNL-SP-7	9	0.4 0.4	TBD	TBD	TBD TBD	TBD TBD	90%
ICP Be	LA-505-159(A) PNL-ALO-101(A)	II/LTRR	LA-505-151 PNL-SP-7	0.3	0.06 0.05	TBD	TBD	TBD TBD	TBD TBD	75%
ICP Bi	LA-505-159(A) PNL-ALO-101(A)	I/LTRR	LA-505-151 PNL-SP-7	39	13 TBD	TBD	TBD	TBD TBD	TBD TBD	90%
ICP Ca	LA-505-159(A) PNL-ALO-101(A)	II/STIR	LA-505-151 PNL-SP-7	1,790	0.03 0.1	TBD	TBD	TBD TBD	TBD TBD	75%
ICP Cd	LA-505-159(A) PNL-ALO-101(A)	I/STIR	LA-505-151 PNL-SP-7	5	0.6 0.4	TBD	TBD	TBD TBD	TBD TBD	90%
ICP Ce	LA-505-159(A) PNL-ALO-101(A)	III/STIR	LA-505-151 PNL-SP-7	3,780	2.1 30	TBD	TBD	TBD TBD	TBD TBD	50%
ICP Co	LA-505-159(A) PNL-ALO-101(A)	TBD/Reg	LA-505-151 PNL-SP-7	TBD	3.7 31	TBD	TBD	TBD TBD	TBD TBD	50%
ICP Cr	LA-505-159(A) PNL-ALO-101(A)	I/STIR	LA-505-151 PNL-SP-7	0.8	0.6 1.9	TBD	TBD	TBD TBD	TBD TBD	90%

Table D-3. Data Quality Objectives and Laboratory Capabilities. (sheet 2 of 10)

Parameter	Preparation WHC PNL	Type/basis	Method WHC PNL	Threshold concentration µg/g	Detection limit µg/g	DQO accuracy	DQO precision	Laboratory accuracy solid liquid	Laboratory precision solid liquid	Completeness
Inorganic metals (µg/g or µg/mL)										
ICP Cr(VI)	LA-504-101(W) PNL-ALO-103(W)	I/LTRR	LA-505-151 PNL-SP-7	0.8	2.1 1.0	TBD	TBD	TBD TBD	TBD TBD	90%
ICP Cr(VI)	LA-504-101 (W) PNL-ALO-103(W)	I/LTRR	LA-265-101 PNL-ALO-227	0.8	5 TBD	TBD	TB	TBD TBD	TBD TBD	90%
ICP Cu	LA-505-159(A) PNL-ALO-101(A)	III/Reg	LA-505-151 PNL-SP-7	TBD	2.3 1.3	TBD	TBD	TBD TBD	TBD TBD	50%
ICP Fe	LA-505-159(A) PNL-ALO-101(A)	I/STIR	LA-505-151 PNL-SP-7	438	1.1 0.7	TBD	TBD	TBD TBD	TBD TBD	90%
CVAA Hg	Direct	II/LTRR	LA-325-102 PNL-ALO-213	0.8	0.005 0.05	TBD	TBD	TBD TBD	TBD TBD	75%
ICP K	LA-505-159(A) PNL-ALO-101(A)	II/CLASS	LA-505-151 PNL-SP-7	4,380	32 65	TBD	TBD	TBD TBD	TBD TBD	75%
ICP La	LA-505-159(A) PNL-ALO-101(A)	III/CLASS	LA-505-151 PNL-SP-7	4,380	2.1 2.4	TBD	TBD	TBD TBD	TBD TBD	50%
ICP Mg	LA-505-149(A) PNL-ALO-101(A)	III/Reg	LA-505-151 PNL-SP-7	TBD	0.02 0.2	TBD	TBD	TBD TBD	TBD TBD	90%
ICP Mn	LA-505-149(A) PNL-ALO-101(A)	I/STIR	LA-505-151 PNL-SP-7	19	0.2 0.2	TBD	TBD	TBD TBD	TBD TBD	90%
ICP Mn(VII)	LA-504-101(W) PNL-ALO-103(W)	TBD/Reg	LA-505-151 PNL-SP-7	TBD	0.2 0.07	TBD	TBD	TBD TBD	TBD TBD	90%
ICP Na	LA-505-149(A) PNL-ALO-101(A)	I/LTRR	LA-505-151 PNL-SP-7	4,380	7.2 28	TBD	TBD	TBD TBD	TBD TBD	90%
ICP Ni	LA-505-149(A) PNL-ALO-101(A)	I/STIR	LA-505-151 PNL-SP-7	39	2.2 3.6	TBD	TBD	TBD TBD	TBD TBD	90%
ICP Pb	LA-505-149(A) PNL-ALO-101(A)	I/STIR	LA-505-151 PNL-SP-7	27	4.1 15	TBD	TBD	TBD TBD	TBD TBD	90%

Table D-3. Data Quality Objectives and Laboratory Capabilities. (sheet 3 of 10)

Parameter	Preparation WHC PNL	Type/Basis	Method WHC PNL	Threshold concentration µg/g	Detection limit µg/g	DQO accuracy	DQO precision	Laboratory accuracy solid liquid	Laboratory precision solid liquid	Completeness
Inorganic metals (µg/g or µg/mL)										
GFAA Pb	None PNL-ALO-101(A)	I/STIR	NONE PNL-ALO-216	27	NA TBD	TBD	TBD	TBD TBD	TBD TBD	90%
ICP Se	LA-505-149(A) PNL-ALO-101(A)	III/Reg	LA-505-151 PNL-SP-7	3	9.5 30	TBD	TBD	TBD TBD	TBD TBD	90%
HYAA GFAA Se	Direct PNL-ALO-101(A)	III/Reg	LA-365-131 PNL-ALO-215	3	0.03 1.0	TBD	TBD	TBD TBD	TBD TBD	90%
ICP Sb	LA-505-159(A) PNL-ALO-101(A)	II/LTRR	LA-505-151 PNL-SP-7	0.9	14 58	TBD	TBD	TBD TBD	TBD TBD	75%
HYAA GFAA Sb	None PNL-ALO-101(A)	II/LTRR	None PNL-ALO-219	0.9	TBD TBD	TBD	TBD	TBD TBD	TBD TBD	75%
ICP Si	LA-505-159(A) PNL-ALO-101(A)	I/CLASS	LA-505-151 PNL-SP-7	134	4.7 4.7	TBD	TBD	TBD TBD	TBD TBD	90%
ICP Sn	LA-505-149(A) PNL-ALO-101(A)	III/Reg	LA-505-151 PNL-SP-7	447	2.2 NA	TBD	TBD	TBD TBD	TBD TBD	50%
ICP Th	LA-505-149(A) PNL-ALO-101(A)	III/Gen	LA-505-151 PNL-SP-7	None	1.8 20	TBD	TBD	TBD TBD	TBD TBD	50%
ICP Tl	LA-504-101(A) PNL-ALO-103(A)	III/Reg	LA-505-151 PNL-SP-7	None	6.6 500	TBD	TBD	TBD TBD	TBD TBD	50%
GFAA Tl	None PNL-ALO-101(A)	III/Reg	None PNL-ALO-220	None	NA 2	TBD	TBD	TBD TBD	TBD TBD	50%
ICP U	LA-505-159(A) PNL-ALO-101(A)	I/LTRR	LA-505-151 PNL-SP-7	20	171 169	TBD	TBD	TBD TBD	TBD TBD	90%
FLUOR U	LA-549-141(F) PNL-ALO-102(F)	I/LTRR	LA-925-106 HTA-4-16	20	0.05 TBD	TBD	TBD	TBD TBD	TBD TBD	90%

Table D-3. Data Quality Objectives and Laboratory Capabilities. (sheet 4 of 10)

Parameter	Preparation WHC PNL	Type/basis	Method WHC PNL	Threshold concentration µg/g	Detection limit µg/g	DQO accuracy	DQO precision	Laboratory accuracy solid liquid	Laboratory precision solid liquid	Completeness
Inorganic metals (µg/g or µg/mL)										
ICP V	LA-505-159(A) PNL-ALO-101(A)	II/LTRR	LA-505-151 PNL-SP-7	49	1.9 0.8	TBD	TBD	TBD TBD	TBD TBD	75%
ICP Zn	LA-505-159(A) PNL-ALO-101(A)	III/Reg	LA-505-151 PNL-SP-7	None	0.3 1.5	TBD	TBD	TBD TBD	TBD TBD	50%
ICP Zr	LA-505-159(A) PNL-ALO-101(A)	I/STIR	LA-505-151 PNL-SP-7	4,100	2.1 1.1	TBD	TBD	TBD TBD	TBD TBD	90%
[a]ICP Al	LA-549-141(F) PNL-ALO-102(F)	Same as above	LA-505-151 PNL-SP-7	Same as above	110 140	TBD	TBD	TBD TBD	TBD TBD	90%
[a]ICP Bi	LA-549-141(F PNL-ALO-102(F)	Same as above	LA-505-151 PNL-SP-7	Same as above	130 130	TBD	TBD	TBD TBD	TBD TBD	90%
[a]ICP Fe	LA-549-141(F) PNL-ALO-102(F)	Same as above	LA-505-151 PNL-SP-7	Same as above	11 20	TBD	TBD	TBD TBD	TBD TBD	90%
[a]ICP Si	LA-549-141(F) PNL-ALO-102(F)	Same as above	LA-505-151 PNL-SP-7	Same as above	47 160	TBD	TBD	TBD TBD	TBD TBD	90%
[a]ICP Th	LA-549-141(F) PNL-ALO-102(F)	Same as above	LA-505-151 PNL-SP-7	Same as above	18 320	TBD	TBD	TBD TBD	TBD TBD	90%
[a]FLUOR U	LA-549-141(F) PNL-ALO-102(F)	Same as above	LA-925-106 HTA-4-16	Same as above	0.05 TBD	TBD	TBD	TBD TBD	TBD TBD	90%
[a]ICP Zr	LA-549-141(F) PNL-ALO-102(F)	Same as above	LA-505-151 PNL-SP-7	Same as above	21 24	TBD	TBD	TBD TBD	TBD TBD	90%

Table D-3. Data Quality Objectives and Laboratory Capabilities. (sheet 5 of 10)

Parameter	Preparation WHC PNL	Type/basis	Method WHC PNL	Threshold concentration µg/g	Detection limit µg/g	DQO accuracy	DQO precision	Laboratory accuracy solid liquid	Laboratory precision solid liquid	Completeness
Inorganic anions (µg/g or µg/mL)										
IC NO_3^-	LA-504-101(W) PNL-ALO-103(W)	I/LTRR	LA-533-105 PNL-ALO-212	94	40	TBD	TBD	TBD TBD	TBD TBD	90%
IC NO_2^-	LA-504-101(W) PNL-ALO-103(W)	I/STIR	LA-533-105 PNL-ALO-212	4	40	TBD	TBD	TBD TBD	TBD TBD	90%
SPEC NO_2^-	LA-504-101(W) PNL-ALO-103(W)	I/STIR	LA-645-101 NA	4	5	TBD	TBD	TBD TBD	TBD TBD	90%
IC F^-	LA-504-101(W) PNL-ALO-103(W)	I/LTRR	LA-533-105 PNL-ALO-212	113	20	TBD	TBD	TBD TBD	TBD TBD	90%
SIE F^-	LA-504-101(W) PNL-ALO-103(W)	I/LTRR	LA-371-135 HWVP-2	113	TBD TBD	TBD	TBD	TBD TBD	TBD TBD	90%
IC SO_4^{-2}	LA-504-101(W) PNL-ALO-103(W)	I/STIR	LA-533-105 PNL-ALO-212	4,330	40	TBD	TBD	TBD TBD	TBD TBD	90%
IC PO_4^{-3}	LA-504-101(W) PNL-ALO-103(W)	I/STIR	LA-533-105 PNL-ALO-212	433	40	TBD	TBD	TBD TBD	TBD TBD	90%
IC Cl^-	LA-504-101(W) PNL-ALO-103(W)	III/GEN	LA-533-105 PNL-ALO-212	114	20	TBD	TBD	TBD TBD	TBD TBD	50%
DIST $Fe(CN)_6^{-4}$ SPEC	None None	Safety $Fe(CN)_6^{-4}$	None None	TBD	TBD	TBD	TBD	TBD TBD	TBD TBD	TBD
DIST CN^- SPEC	Direct Direct	I/LTRR	LA-695-101 PNL-ALO-270	0.4	1 1	TBD	TBD	TBD TBD	TBD TBD	90%
pH OH^-	Direct	I/STIR	LA-212-103 PNL-ALO-225	440	NA	TBD	TBD	TBD TBD	TBD TBD	90%
TIC CO_3^{-2}	LA-504-101(W) PNL-ALO-103(W)	I/STIR	LA-622-102 7-40.7	3340	250 500	TBD	TBD	TBD TBD	TBD TBD	90%
DIST S^{-2} SIE	Direct Direct	REG	None None	500	TBD	TBD	TBD	TBD TBD	TBD TBD	50%
Other NH_3	LA-504-101(W) PNL-ALO-103(W)	III/REG	LA-634-102 PNL-ALO-226	100	90 5	TBD	TBD	TBD TBD	TBD TBD	50%

Table D-3. Data Quality Objectives and Laboratory Capabilities. (sheet 6 of 10)

Parameter	Preparation WHC PNL	Type/basis	Method WHC PNL	Threshold concentration µg/g	Detection limit µg/g	DQO accuracy	DQO precision	Laboratory accuracy solid liquid	Laboratory precision solid liquid	Completeness
Organic analyses (µg/g or µg/mL)										
TOC	LA-501-101(W) PNL-ALO-103(W)	Screen	LA-344-105 7-40.7	None	100	TBD	TBD	TBD TBD	TBD TBD	90%
EOX/TOX	PNL-ALO-320	Screen	None PNL-ALO-320	None	NA TBD	TBD	TBD	TBD TBD	TBD TBD	75%
Complexants (µg/g or µg/mL)										
EDTA	LA-504-101(W) PNL-ALO-103(W)	I/LTRR	None None	0.09	TBD	TBD	TBD	TBD TBD	TBD TBD	90%
HEDTA	LA-504-101(W) PNL-ALO-103(W)	I/STIR	None None	438	TBD	TBD	TBD	TBD TBD	TBD TBD	90%
Citrate	LA-504-101(W) PNL-ALO-103(W)	I/CLASS	None None	4380	TBD	TBD	TBD	TBD TBD	TBD TBD	90%
Hydroxy-acetate	LA-504-101(W) PNL-ALO-103(W)	II/CLASS	None None	4,380	TBD	TBD	TBD	TBD TBD	TBD TBD	75%
Oxalate	LA-504-101(W) PNL-ALO-103(W)	III/CLASS	None None	322	TBD	TBD	TBD	TBD TBD	TBD TBD	50%
VOAs	None PNL-ALO-335	REG	None PNL-ALO-335	None	TBD[b]	TBD	TBD	TBD TBD	TBD TBD	75%
Semi-VOAs	None PNL-ALO-120	REG	None PNL-ALO-345	None	TBD[b]	TBD	TBD	TBD TBD	TBD TBD	75%

Table D-3. Data Quality Objectives and Laboratory Capabilities. (sheet 7 of 10)

Parameter	Preparation WHC PNL	Type/basis	Method WHC PNL	Threshold concentration nCi/g	Detection limit nCi/g	DQO accuracy	DQO precision	Laboratory accuracy solid liquid	Laboratory precision solid liquid	Completeness
Radionuclides (μCi/g or μCi/mL)[d]										
LS ^{3}H	LA-504-101[W] PNL-ALO-103[W]	TBD	LA-218-113 PNL-SP-30	TBD	TBD	TBD	TBD	TBD TBD	TBD TBD	90%
LS ^{14}C	LA-504-101[W] PNL-ALO-103[W]	I/LTRR	LA-348-104 PNL-ALO-442	0.6	0.05	TBD	TBD	TBD TBD	TBD TBD	90%
^{63}Ni	LA-505-159[A] PNL-ALO-101[A]	I/CLASS	None PNL-ALO-439	58	TBD	TBD	TBD	TBD TBD	TBD TBD	90%
LS ^{79}Se	LA-549-141[F] PNL-ALO-102[F]	III/LTRR	LA-365-135 PNL-ALO-444	TBD	TBD	TBD	TBD	TBD TBD	TBD TBD	75%
Beta ^{90}Sr	LA-549-141[F] PNL-ALO-102[F]	I/STIR	LA-220-102 PNL-ALO-433	233	4	TBD	TBD	TBD TBD	TBD TBD	90%
LEPS ^{93}Nb	LA-549-141[F] PNL-ALO-102[F]	II/LTRR	None None	TBD	TBD	TBD	TBD	TBD TBD	TBD TBD	75%
LS and beta ^{99}Tc	LA-549-141[F] PNL-ALO-102[F]	I/LTRR	LA-438-101 PNL-ALO-432	0.1	2.5	TBD	TBD	TBD TBD	TBD TBD	90%
LEPS ^{129}I	LA-549-141[F] PNL-ALO-102[F]	I/LTRR	LA-378-103 PNL-ALO-454	0.002	10	TBD	TBD	TBD TBD	TBD TBD	90%
GEA ^{137}Cs	LA-549-141[F] PNL-ALO-102[F]	I/LTRR	LA-548-121 PNL-ALO-451	382	160	TBD	TBD	TBD TBD	TBD TBD	90%
U-Isotopic ^{235}U ^{238}U	LA-549-141[F] PNL-ALO-102[F]	I/LTRR I/LTRR	None PNL-ALO-455	0.004 0.007	3.5E-10	TBD	TBD	TBD TBD	TBD TBD	90%
Alpha ^{237}Np	LA-549-141[F] PNL-ALO-102[F]	II/LTRR	LA-933-141 PNL-ALO-425	0.3	1.6	TBD	TBD	TBD TBD	TBD TBD	75%

Table D-3. Data Quality Objectives and Laboratory Capabilities. (sheet 8 of 10)

Parameter	Preparation WHC PNL	Type/basis	Method WHC PNL	Threshold concentration nCi/g	Detection limit nCi/g	DQO accuracy	DQO precision	Laboratory accuracy solid liquid	Laboratory precision solid liquid	Completeness
Radionuclides (μCi/g or μCi/mL)[c]										
Alpha ^{238}Pu $^{239/240}Pu$	LA-549-141(F) PNL-ALO-102(F)	I/STIR I/LTRR	LA-503-156 PNL-ALO-423	0.3 0.3	0.8	TBD	TBD	TBD TBD	TBD TBD	90%
Pu Isotopic ^{240}Pu ^{241}Pu	LA-549-141(F) PNL-ALO-102(F)	I/LTRR I/STIR	None PNL-ALO-455	0.3 10	0.8	TBD	TBD	TBD TBD	TBD TBD	90%
Alpha ^{241}Am ^{244}Cm	LA-549-141(F) PNL-ALO-102(F)	I/STIR II/STIR	LA-503-156 PNL-ALO-424	0.3 0.3	0.8	TBD	TBD	TBD TBD	TBD TBD	90%
Physical measurement										
Wt% H_2O	Direct	NA	LA-564-101 PNL-ALO-504	NA	NA	TBD	TBD	TBD TBD	TBD TBD	90%
Bulk density g/cm^3	Direct	NA	T038 WHC-053-1	NA	NA	TBD	TBD	TBD TBD	TBD TBD	90%
Penetrometer psi	Direct	NA	T038 PNL-ALO-506	Dilatent Cohesive	0-10 psi >100 psi	$\pm$10%	$\pm$10%	TBD TBD	TBD TBD	90%
Particle size μm	Direct	NA	T044 2-50.3	0.1-20μm	TBD	<5%	<5%	TBD TBD	TBD TBD	90%
Shear stress/ shear rate	Direct	NA	None WHC-053-1	NA	NA	$\pm$15%	$\pm$15%	TBD TBD	TBD TBD	90%
Absolute viscosity Cp	Direct	NA	None WHC-053-1	40-20 x 10^6 cp	TBD	$\pm$10%	$\pm$10%	TBD TBD	TBD TBD	90%
Shear strength dynes/cm	Direct	NA	None WHC-053-1	0-1x10^5 dynes/cm	TBD	$\pm$10%	$\pm$10%	TBD TBD	TBD TBD	90%

Table D-3. Data Quality Objectives and Laboratory Capabilities. (sheet 9 of 10)

Parameter	Preparation WHC PNL	Type/basis	Method WHC PNL	Threshold concentration µg/g	Detection limit µg/g	DQO accuracy	DQO precision	Laboratory accuracy solid liquid	Laboratory precision solid liquid	Completeness
Physical measurements										
Porosity	Direct	NA	None None	NA	TBD	±10X	±10X	TBD TBD	TBD TBD	90%
Compressive strength psi	Direct	NA	None None	NA	TBD	±10X	±10X	TBD TBD	TBD TBD	90%
Characteristics										
X-ray Diffraction	Direct	Safety $Fe(CN)_6^{-4}$	LA-507-151 LA-507-152 None	TBD	TBD	TBD	TBD	TBD	TBD	TBD
Polarized Light Microscopy	Direct	Safety $Fe(CN)_6^{-4}$	RHO-RE-ST-28P None	TBD	TBD	TBD	TBD	TBD	TBD	TBD
Corrositivity pH	Direct	Reg	LA-212-103 PNL-ALO-225	pH-12	NA	±0.5%	±0.5%	TBD TBD	TBD TBD	90%
Reactivity CN (µg/g) S T°, Cal. DSC	Direct	Reg	None None LA-514-113 RDS-TA-1	 250 µg/g 500 µg/g NA	 Exotherm	NA	NA	NA NA	NA NA	90%
Ignitibility Flash Pt DSC	Direct	Reg	TBD LA-514-113 RDS-TA-1	60 °C NA	NA Positive exotherm	NA NA	NA NA	NA NA	NA NA	90%
[d]TCLP As mg/L Ba Cd Cr Pb Hg Se Ag	Extract	Reg	None None	5 mg/L 100 mg/L 1 mg/L 5 mg/L 5 mg/L 0.2 mg/L 1.0 mg/L 5.0 mg/L	As-.04 mg Ba-.01 L Cd - .01 Cr - .02 Pb - .09 Hg - .01 Se - .04 Ag - .01	TBD	TBD	TBD TBD	TBD TBD	90%
PA-Solubility Metals Anions Rads Org	TBD	PA	TBD	TBD	TBD	TBD	TBD	TBD TBD	TBD TBD	90%

Table D-3. Data Quality Objectives and Laboratory Capabilities. (sheet 10 of 10)

Parameter	Preparation WHC PNL	Type/basis	Method(e) WHC PNL	Threshold concentration µg/g	Detection limit µg/g	DQO accuracy	DQO precision	Laboratory accuracy solid liquid	Laboratory precision solid liquid	Completeness
Thermal Output	Direct	Safety High Heat	None None	TBD	TBD	TBD	TBD	TBD	TBD	TBD
Coefficient of Thermal Expansion	Direct	Safety High Heat	None None	TBD	TBD	TBD	TBD	TBD	TBD	TBD
Specific Heat	Direct	Safety High Heat	None None	TBD	TBD	TBD	TBD	TBD	TBD	TBD
Thermal Conductivity	Direct	Safety High Heat	None None	TBD	TBD	TBD	TBD	TBD	TBD	TBD
Thermal Conductivity of Frozen Sludge	Direct	Safety High Heat	None None	TBD	TBD	TBD	TBD	TBD	TBD	TBD
Deliquescence	Direct	Safety Gas	None None	TBD	TBD	TBD	TBD	TBD	TBD	TBD

Footnotes:

CLASS = waste classification.Semi-VOA=semi-volatile organic analysis.
CVAA = cold vapor atomic absorption spectro.LS=liquid scintillation counting.
DIST = distillation.LTRR=long term release risk.
EOX/TOX = extractable or total organic halide.NA=not applicable.
FLUOR = fluorimetry.REG=regulatory interest.
GEA = gamma energy analysis.SIP=selective ion electrode.
GFAA = grafite furnace atomic absorption.SPEC=spectrophotometric.
HYAA = hydride atomic absorption spectroscopy.STIR=short term intruder risk.
IC = ion chromatography.TBD=to be determined.
LEPS = low energy photon spectroscopy.TCLP=toxicity characteristic leaching pro.
TOC = total organic carbon(W)=Water.
VOA = volatile organic analysis.(F)=Fusion.
(A) = Acid.

[a]Because of acid solubility properties the following metals may be determined more accurately by analysis of a sample prepared by fusion dissolution. However, the detection limits for these metals in the fused sample will be significantly higher because of the large dilutions required to analyze fused samples by ICP. Therefore, ICP values from fusion should only be used if they are greater than acid results and significantly above fusion detection limits.

[b]Detection limits for organics will depend on organic clean up methods in development and on the matrix.

[c]Detection limits are based on normal preparation method and assumes count times and backgrounds based on Table E-1. Note accurate detection limits for each lab will be will be provided in the future.

[d]Detection limits for TCLP will be 5 times lower than acid digestion values for ICP and AA methods because dilution factor differences.

NOTE: A list of current procedures and revisions shall be maintained by each labortory and operation.

Table D-4. Assessment of Analytical Detection Limits.

Analyte	DL <CTL	DL <0.1CTL	DL <0.01CTL	Basis
As	Y-HYAA	N	N	LTRR
Ag	Y-ICP	Y-ICP	N	STIR
Al	Y-ICP	N	N	STIR
Ba	Y-ICP	Y-ICP	N	STIR
Be	Y-ICP	N	N	LTRR
Bi	Y-ICP	N	N	STIR
Ca	Y-ICP	Y-ICP	Y-ICP	STIR
Cd	Y-ICP	Y-ICP	N	STIR
Ce	Y-ICP	Y-ICP	Y-ICP	STIR
Cr	Y-ICP[a]	N	N	STIR
Fe	Y-ICP	Y-ICP	N	CLASS
Hg	Y-CVAA	Y-CVAA[a]	Y-CVAA[a]	LTRR
K	Y-ICP	Y-ICP	Y-ICP[a]	CLASS
La	Y-ICP	Y-ICP	Y-ICP	CLASS
Mn	Y-ICP	Y-ICP	Y-ICP	STIR
Na	Y-ICP	Y-ICP	Y-ICP	CLASS
Ni	Y-ICP	Y-ICP	N	CLASS
Pb	Y-ICP	N[b]	N	STIR
Se	Y-HYAA Y-GFAA	Y-HYAA	N	STIR
Sb	N[b]	N	N	LTRR
Si	Y-ICP	Y-ICP	N	STIR
Sn	Y-ICP	Y-ICP	Y-ICP	STIR
U	Y-FLUOR	Y-FLUOR	Y-FLUOR	LTRR
V	Y-ICP	Y-ICP	Y-ICP	LTRR
Zr	Y-ICP	Y-ICP	Y-ICP	STIR
NO_3^-	Y-IC	N	N	LTRR
NO_2^-	N	N	N	LTRR

Table D-4. Assessment of Analytical Detection Limits.

Analyte	DL <CTL	DL <0.1CTL	DL <0.01CTL	Basis
F^-	Y-IC	Y-IC	N	LTRR
SO_4^{-2}	Y-IC	Y-IC	Y-IC	STIR
PO_4^{-3}	Y-IC	Y-IC	N	STIR
Cl^-	Y-IC	Y-IC	N	LTRR
CN^-	N	N	N	LTRR
CO_3^{-2}	Y-TIC	Y-TIC[a]	N	STIR
NH_3	Y-SIE	Y-SIE[c]	N	REG
^{14}C	Y-LS	Y-LS	N	LTRR
^{90}Sr	Y-Beta	Y-Beta	N	STIR
^{99}Tc	N	N	N	LTRR
^{129}I	N	N	N	LTRR
^{137}Cs	Y-GEA	N	N	CLASS
^{235}U	Y-FLUOR	Y-FLUOR	Y-FLUOR	LTRR
^{238}U	Y-FLUOR	Y-FLUOR	Y-FLUOR	LTRR
^{237}Np	N	N	N	CLASS
^{238}Pu ^{239}Pu ^{240}Pu	N	N	N	CLASS
^{241}Am ^{244}Cm	N	N	N	CLASS

Note: Assessment is based on preliminary estimates of analytical detection limits.

[a]PNL detection limit above CTL.

[b]Based only on ICP.

[c]222-S Lab detection limit above CTL.

Y = Yes-analytical technique

N = No

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5.0 SAMPLING PROCEDURES

A discussion of the SST sampling procedures has been provided in Section 3.2 of the Waste Characterization Plan. The samples are taken by the Tank Farm Operations group in accordance with Plant Operating Procedure (POP) TO-020-450 "Perform Core Sampling." The waste characterization plan describes the sampling equipment and the problems encountered with preservation and holding times when analyzing highly radioactive samples. The sampling POP identifies records taken during sampling and initiates the chain-of-custody for samples. Samples are identified by a unique number. Samples shall not be allowed to stand in the field for over 48 h before shipping to the laboratory. The samples are shipped to the Hanford Site laboratories in accordance to POP TO-080-090 "Ship Core Samples." The procedure also addresses the completion of the chain-of-custody form and obtaining the necessary signatures for sample receipt. The sampling team is responsible for documenting any problems and procedural changes affecting the validity of the sample in a field notebook. Copies of the pages describing the problems and identifying the questionable samples (ID numbers) shall be forwarded to the laboratory for addition to the data package for that sample.

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6.0 SAMPLE CUSTODY

The chain-of-custody form is initiated by the sampling team as described in POPs TO-020-450 and TO-080-090. The sample is shipped in a cask, and sealed with a paper seal containing the information shown below:

WASTE TANK SAMPLE SEAL

Supervisor _____ Sample No. _____

Date of Sampling _____ Time of Sampling _____

Shipment No. _____ Serial No. _____

Because of the cask size and truck limitations, only three samples can be shipped at a time. The sealed and labeled samples are shipped to the laboratory along with the chain-of-custody. An example of the field chain-of-custody form is shown in Figure D-1. Examples of forms in this document may not be the most recent version; therefore, the original and most recent documentation shall be referred to for any changes.

The receipt and control of samples in Westinghouse Hanford 222-S Laboratory and PNL 325 Laboratory are described in the laboratory QAPjPs (WHC-SD-CP-QAPP-002, QAPjP No. SA-001) and laboratory operating procedures LO-090-101 and PNL-ALO-010, respectively. The 325 Laboratory uses two additional internal chain-of-custody forms (Figures D-2 and D-3) whereas the 222-S Laboratory uses traveler cards and a sample checkout list (Figures D-4 and D-5) for tracking sample custody in the laboratory. Both laboratories are located in secured areas that have controlled access and that require photo identification (badging) to be permitted in the area.

A portion of the sample will be saved as an archive sample until the data have been analyzed to allow verification of questionable results and to carry-out method development. Archived samples will not be disposed of without concurrence of the Tank Waste Characterization Technology Section.

6.1 SAMPLE BREAKDOWN AND SUBSAMPLING

After the samples are received in the laboratory they are extruded, physical observations are recorded, and subsamples taken for the other parameters. Table D-5 provides a list of the PNL and Westinghouse procedures covering these operations. These procedures include homogenization and compositing operations.

Figure D-1. Field Chain-of-Custody Record.

CHAIN-OF-CUSTODY RECORD FOR CORE SAMPLING

) Shipment Number _____ (2) Sample Number _____ (3) Supervisor* _____

) Tank _____ (5) Riser _____ (6) Segment _____ (7) Core _____ (8) Cask Serial Number _____

ıdiation Survey Data	(9) FIELD	(26) LABORATORY
Over Top Dose Rate		
Side Dose Rate		
Bottom Dose Rate		
Smearable Contamination	(alpha)	(alpha)
	(beta gamma)	(beta gamma)
	RPT* (Signature)	RPT* (Signature)

(10) Shipment Description

A. Work Package Number
B. Cask Seal Number
C. Sampler Number Used
D. Date and Time Sampler Unseated
E. Expected Liquid Content
F. Expected Solid Content
G. Dose Rate Through Drill String
H. Expected Sample Length

1) INFORMATION *(Include statement of laboratory tests to be performed)*

2) Field Comments:

(27) Laboratory Comments

(13) POINT OF ORIGIN	(14) SENDER NAME	(16) DATE RELEASED	(18) DESTINATION	(20) RECIPIENT NAME	(22) DATE RECEIVED
	(15) SENDER SIGNATURE*	(17) TIME RELEASED		(21) RECIPIENT SIGNATURE*	(23) TIME RECEIVED

(19) Seal Intact Upon Release?	(24) Seal Intact Upon Receipt?	(25) Seal Data Consistent with this Record?		
		Shipment No	Cask Seal No	Sample No
☐ Yes ☐ No	☐ Yes ☐ No	☐ Yes ☐ No	☐ Yes ☐ No	☐ Yes ☐ No

ISTRIBUTION White - Office of Sample Management Yellow - Recipient of Sample
Pink - Process Engineering [illegible] Goldenrod - Tank Farm Operations [illegible]

Figure D-2. 325-A Hot Cell Chain-of-Custody.

Chain of Custody Number _____

325-A HOT CELL CHAIN-OF-CUSTODY

DATE OF TRANSFER _____

SENDER _____

RECEIVER _____

WHC SAMPLE IDENTIFICATION NUMBER _____

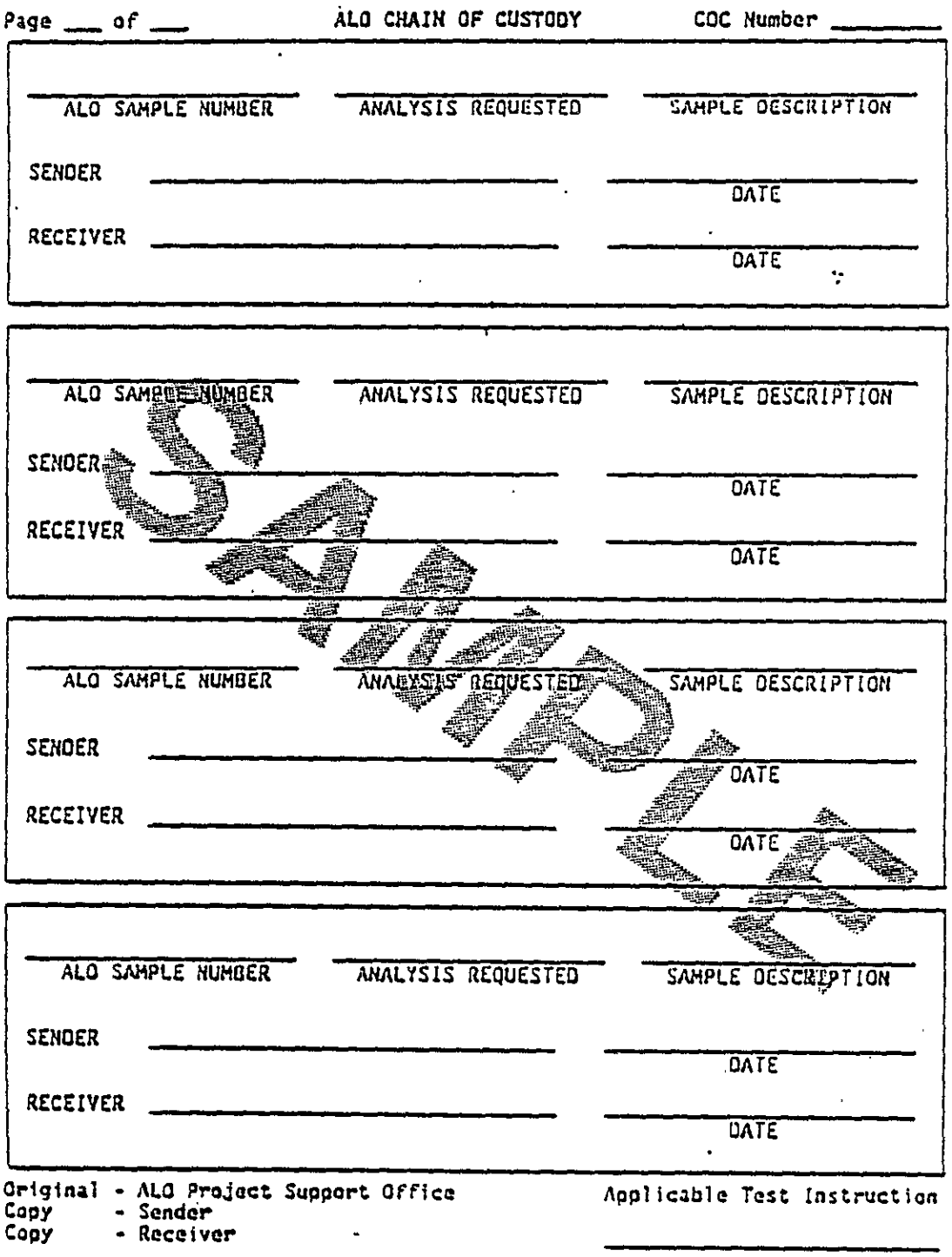
ALO Sample Number 325-A Hot Cell Sample Identification

90-

SAMPLE

Original - ALO Project Support Office
Copy - 325-A Hot Cell
Copy - 325-B Hot Cell

Figure D-3. ALO Chain-of-Custody.



Page ___ of ___ ALO CHAIN OF CUSTODY COC Number _____

ALO SAMPLE NUMBER | ANALYSIS REQUESTED | SAMPLE DESCRIPTION

SENDER _____ DATE _____

RECEIVER _____ DATE _____

ALO SAMPLE NUMBER | ANALYSIS REQUESTED | SAMPLE DESCRIPTION

SENDER _____ DATE _____

RECEIVER _____ DATE _____

ALO SAMPLE NUMBER | ANALYSIS REQUESTED | SAMPLE DESCRIPTION

SENDER _____ DATE _____

RECEIVER _____ DATE _____

ALO SAMPLE NUMBER | ANALYSIS REQUESTED | SAMPLE DESCRIPTION

SENDER _____ DATE _____

RECEIVER _____ DATE _____

Original - ALO Project Support Office
Copy - Sender
Copy - Receiver

Applicable Test Instruction _____

Figure D-4. 222-S Traveler Card.

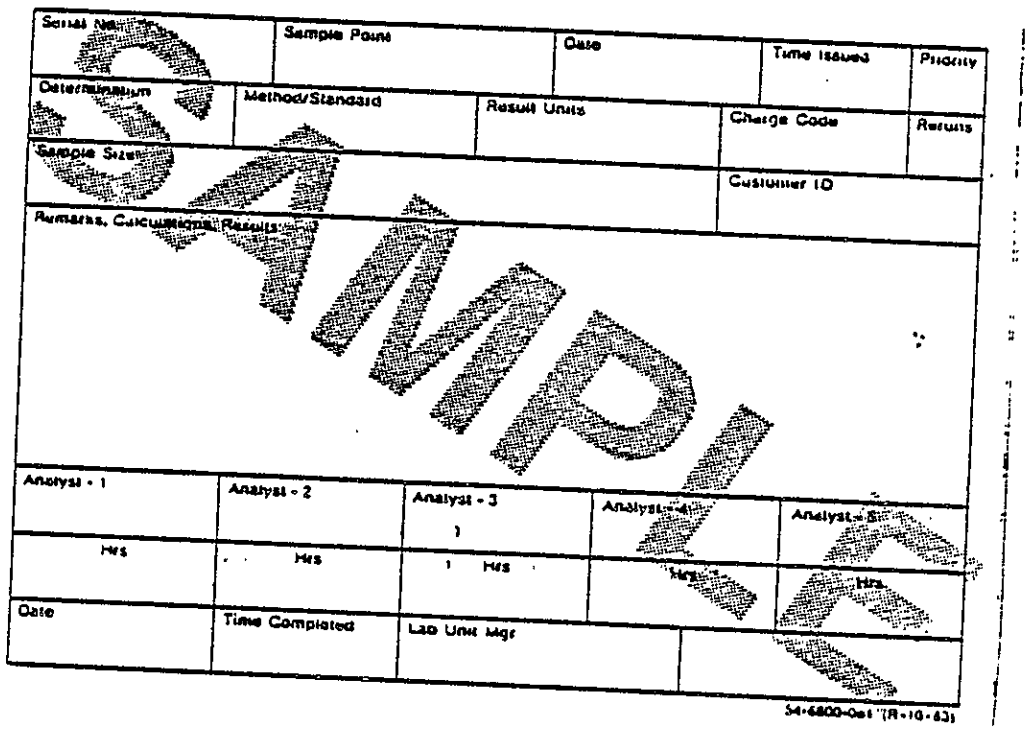
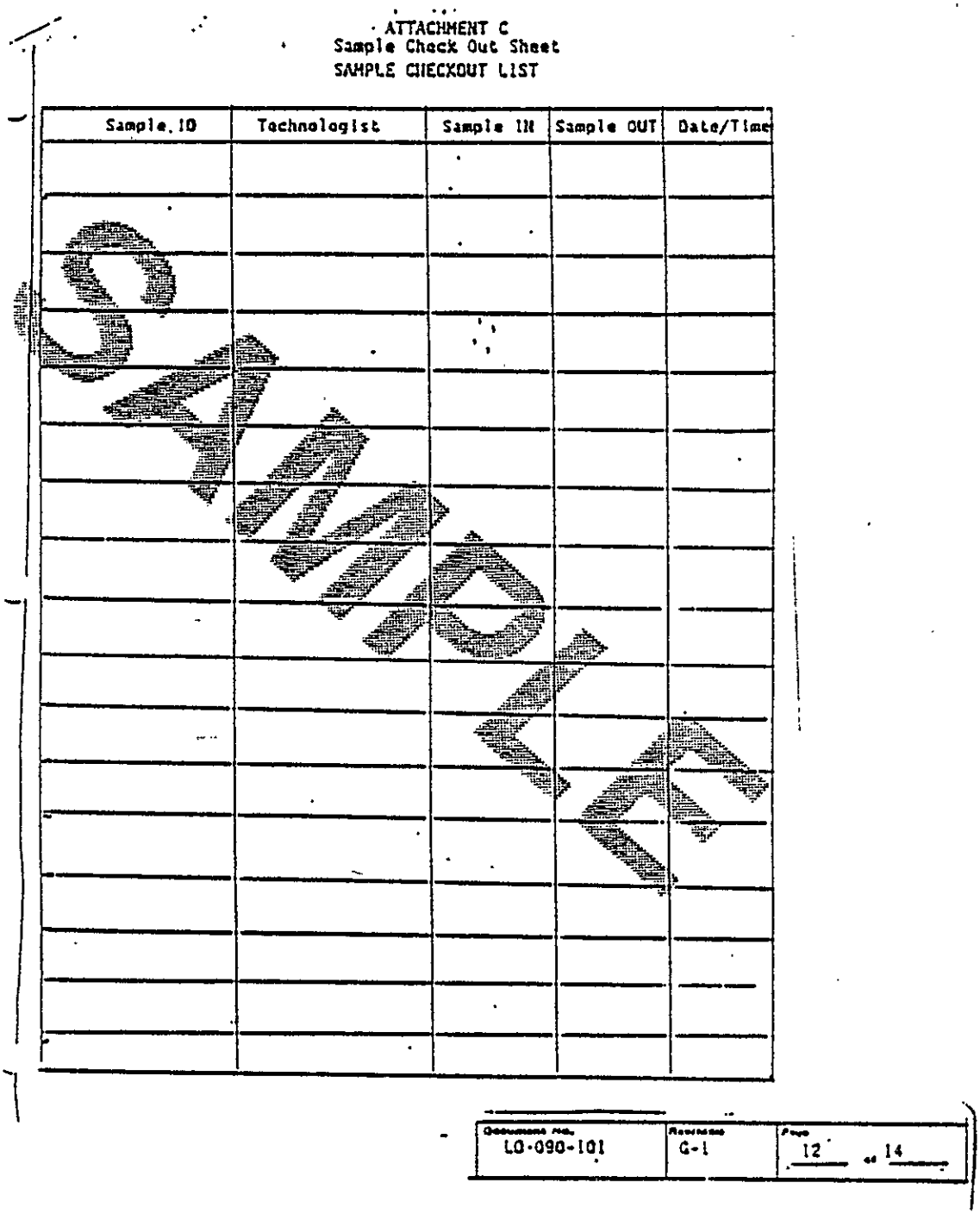


Figure D-5. Sample Checkout List.



ATTACHMENT C
Sample Check Out Sheet
SAMPLE CHECKOUT LIST

Sample ID	Technologist	Sample IN	Sample OUT	Date/Time

Document No.	Revision	Page
LO-090-101	G-1	12 of 14

Table D-5. Sample Breakdown Procedures.

Westinghouse Hanford Process Chemistry Laboratories Desk Instructions	
Number	Title
T038 A-00 712F	Sampler Extruder Operations and Sample Breakdown
T039 A-00 712F	Core Sample Cask Disassembly and Sampler Loading in IE-2 Hot Cell
T040 A-00 712F	Core Sampler Cask Receipt
T046 A-00 712F	Segment Breakdown and Analytical Sample Schedule
Pacific Northwest Laboratory Procedure	
PNL-ALO-010 (Rev 0)	325 Laboratory Single-Shell Tank Sample Receiving and Subsample/Analysis Numbering System
325-A-29 (Rev 0)	Receiving of Waste Tank Samples in Onsite Transfer Cask
PNL-ALO-130 (Rev 0)	Receipt and Inspection of SST Samples
325-EXT-1 (Rev 0)	Receipt and Extrusion of Core Samples at 325A Shielded Facility
PNL-ALO-135 (Rev 0)	Homogenization of Solutions, Slurries, and Sludges

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7.0 CALIBRATION PROCEDURES AND FREQUENCY

Calibration frequency requirements for the WHC and PNL laboratories are listed in Table D-6. Detailed calibration procedures for methods and instruments shall be in accordance with procedures listed in Table D-3. These same procedures describe the standards and their sources. Sources for purchased standards and standardization methods and results shall be documented. Any system whose calibration verification standard is outside its 3σ limit shall be checked more thoroughly and recalibrated as necessary. Samples analyzed on systems out of calibration shall be reanalyzed. However, if data must be obtained from systems with performance outside the calibration limit, the data shall be flagged, the calibration problem described, and the recovery for the calibration check standard provided in data reports. An example where operating a system out of calibration may be acceptable is when a Type III analyte is out of control on the ICP and recalibration and rerunning the sample for this low priority analyte is not justified.

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8.0 ANALYTICAL PROCEDURES

The analysis of all SST waste characterization samples and related data shall be implemented in accordance with WHC and PNL procedures listed in Table D-3 as specified in the laboratory statement of work (SOW) initiated by the OSM. These procedures shall meet the specific requirements described in the laboratory QA project plans (WHC 1989, PNL 1989). The procedures applicable to SST waste characterization are identified in Table D-3. Laboratories and sampling operations shall maintain a list of all current procedures and latest revisions that are being used for SST waste characterization.

A summary description of the deviations from SW 846 test methods are described in Section 5.0 of the Waste Characterization Plan. Such deviations from SW 846 have been incorporated in those analytical procedures listed in Table D-3. Analysis of SST waste materials shall be in accordance with procedures listed in Table D-3 and include applicable quality control checks described in Section 10.0 with a description of the basis for establishing detection limits and for performing and verifying calibrations.

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9.0 DATA REDUCTION, VALIDATION, AND REPORTING

Data reduction, validation and reporting policies and requirements are described in the 222-S Laboratory and 325 Laboratory QA project plans (WHC 1989, PNL 1989). Data reduction methods and equations shall be included in each analytical procedure.

Criteria to validate data are discussed in the two QA project plans governing this work. The following are some of the validation areas common to the methods.

1. Sample recovery (percent of sample which is obtained based on what was expected) will be used to evaluate the sampling process. Partial or empty segments will increase the uncertainty in the representativeness of the sample and limit the scope of testing that can be done. A 75% or greater recovery of the sample is considered to be an acceptable criteria.

2. Sample receipt and tracking documents shall be complete to ensure that the sample has been shipped and processed in a timeframe and manner that would not introduce significant errors. Meeting holding times and preservation (refrigeration) requirements for constituents in highly radioactive samples will not be possible during Phase I. However, efforts will be made to minimize analysis times for these constituents.

3. All data shall be gathered with systems calibrated in accordance with applicable procedures. Data reported with out of control calibration conditions shall be flagged.

4. Field, hot cell, and analytical method blanks will be evaluated to ensure contamination is not a factor in the final results. Data collected or reported under high blank conditions will be identified.

5. The relative percentage differences for duplicate analyses of samples will be used to evaluate homogenization and core compositing procedures. Replicate analyses of digestions will be used to evaluate procedure performance. Data outside the control limits will be identified in the data reports to OSM and explanations of the data provided.

6. Matrix spike recoveries will be used to evaluate the applicability of the procedure to the matrix and will be reported for applicable constituents. Data considered to be questionable because of poor spike recoveries will be flagged.

7. Detection limits and the basis for determining the limits for each analyte will be reported for comparison to DQOs.

8. Estimates of precision and accuracy information for each parameter will be collected for the sample results and laboratory control standards. This information will be used to verify the analytical system performance based on control limits established from historical data.

Figures D-6 and D-7 summarize the data flow through the 222-S and 325 Laboratories. Data packages will be prepared for each segment analyzed in the core and the core composite. The data package shall include the following general items:

- Identification of procedures used
- Summary of results
- Chain-of-custody records
- Field sampling observations
- Hot cell breakdown and preparation observations
- Analytical results for inorganics, radionuclides, organics, physical analyses and characteristic tests
- Quality control results for each test
- Calibration and standards documentation for each test
- Supporting information.
  - Chromatograms
  - Interelement corrections
  - Detection limits
  - Worksheets and travelers.
  - Laboratory notebook records of procedure deviations or problems.

The report also shall identify and discuss any problems encountered in the sampling, breakdown, and analysis operations and the impact these problems may have had on the results. The report shall be prepared and approved as described in laboratory QAPjPs and procedures.

The data reduction equations for analysis of each parameter are described in each procedure.

The principal criteria that will be used to validate data integrity during collection is summarized in Table D-6 and described in Section 10.0 of this QAPjP. The primary criteria are: calibration verification, blank

Figure D-6. Data and Information Flow at 222-S Laboratory.

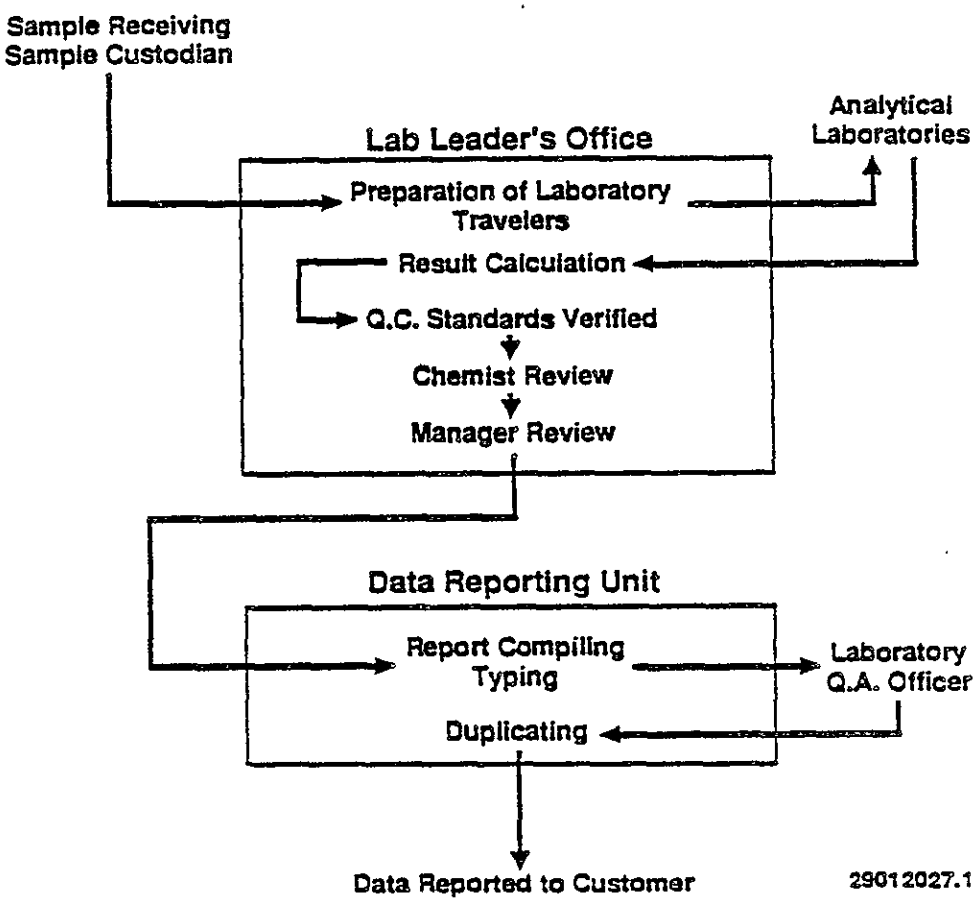
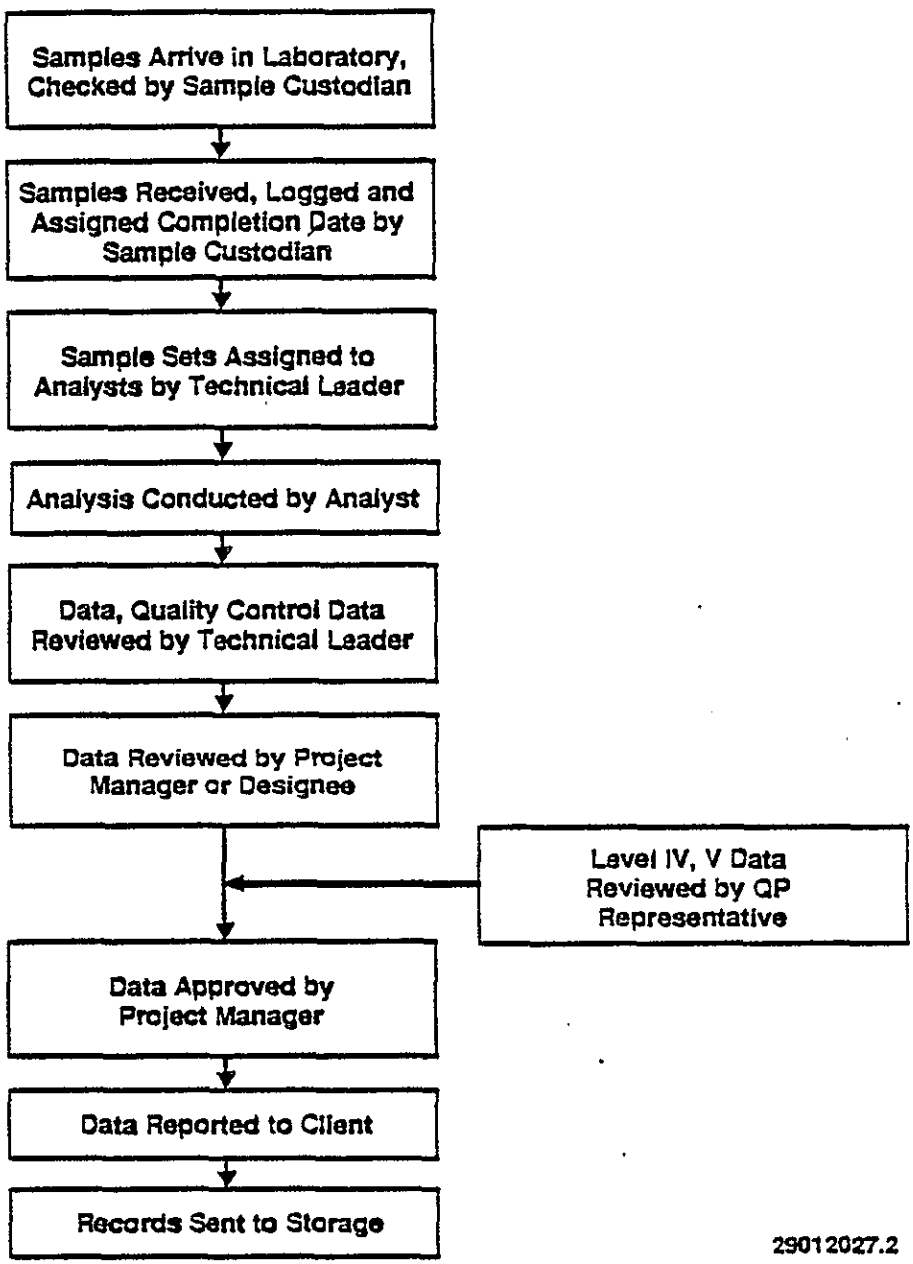


Figure D-7. Data and Information Flow at 325 Laboratory.



evaluations, laboratory control standard performance, duplicate analyses results, matrix spike results, and detection limits. In addition, tests to evaluate the homogenization and compositing procedure reproducibility and performance will be performed for each tank. Criteria for validating data packages is described in Sections 2.0 and 2.2 of the Westinghouse Hanford Office of Sample Management, *Sample Management Administration Manual*, WHC-CM-5-3.

Data for the principal criteria shall be collected and analyzed to measure the standard deviation so that $\pm 3\sigma$ control limits can be established. For calibration verification, laboratory control standards and matrix spikes a mean recovery and 3σ limits shall initially be determined and updated at least every 3 months (if the program has been active) or after the analysis of each batch of tanks. After a sufficient database has been established control limits may be updated less frequently. For duplicates, matrix spike duplicates, homogenization tests, and core composite tests historical information will be collected for each tank and the average relative percent difference in results and its $\pm 3\sigma$ limit shall be used to identify unusual conditions when testing new tanks. Outliers will be identified by using control charts, tabulated data or computer analysis of data. Outliers will be flagged and the cause of the outlier evaluated and documented by reanalysis of the sample, standards, or other appropriate action depending on the analysis as discussed in the next Section 10.0.

Control of software for data reduction and reporting are addressed in the Defense Operations Division Laboratories, operating instructions (WHC-CM-5-4) and in 222-S Laboratory procedures LC-400-005, Laboratory Computer Control and LC-400-006 Spreadsheet Documentation Guidelines. The PNL procedures for calculating ICP and ion chromatography results are provided in the procedure PNL-ALO-218, ICP/IC Data Calculations. General computer and software control requirements are described in the PNL Quality Assurance Manual PNL-MA-70.

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10.0 INTERNAL QUALITY CONTROL

10.1 GENERAL DISCUSSION OF SINGLE-SHELL TANK QUALITY CONTROL OBJECTIVES

Quality control (QC) guidelines are described in the 222-S and 325 Laboratories QA project plans (WHC 1989, PNL 1989). Laboratory quality control requirements are based on requirements of the SW-846 and EPA Contract Laboratory Program (CLP) procedures. The frequency of the QC checks for these procedures are specified for a batch or set of samples. Batch sizes for highly radioactive samples may be small (1 to 5) compared to nonradioactive samples (10 to 20) because of the need to control personnel exposure and contamination. This can result in a high proportion of QC checks. The requirements described in this section apply to the characterization of all SSTs unless modified in the test plan for a specific set of tanks.

To control cost and exposure but still ensure that adequate control data is obtained, the test plan for sampling and analysis of each tank or a group of tanks will include QC elements that may redefine the number of duplicates and other QC checks that are described in this section. Since the number of samples (segments) will vary with waste depth, and the number of cores needed will depend on decision requirements being developed, the total number of analyses could vary significantly between tanks, particularly if a large number of parameters must be determined on segments. The test plan will be designed on the premise that all the samples (segments and core composites) from the tank constitute a tank batch or set of samples from the tank. Many QC checks such as calibration, laboratory control standards, and blanks are used to support the results for the analytical batch and must be performed each time samples are analyzed. Other QC checks such as duplicates and matrix spikes are performed to measure the performance of methods on a particular sample matrix or tank batch. The objectives of the test plan must be: (1) to ensure that the analytical system is in control during measurements, (2) that sufficient duplicates are performed on each tank to permit error estimates that meet DQOs, and (3) that sufficient matrix spikes are performed to ensure that method performance is adequate for that matrix to meet DQOs. Table D-6 has been prepared to summarize these checks for each parameter and establish minimum QC guidelines for the SST characterization. A discussion of the QC guidelines for each parameter is provided. These guidelines may be modified in each test plan or increased by operations when conditions indicate a higher level of QC may be required. Quality control charts, tabulated data and reports will be used to evaluate the performance of the sampling and analysis system of each tank and between tanks. The following paragraphs provide a general discussion of the column headings to Table D-6.

Calibration verification and laboratory control standards may be used interchangeably for some parameters. Calibration verification standards are independent standards (different source than those used for calibration) that are not normally processed through the preparation steps but are used only for instrument control. Laboratory control standards, on the other hand, shall be carried through both the preparation and analysis steps and are used to monitor the performance of the analytical system for the entire method.

Preparing stable and accurate standards in SST waste matrices is extremely difficult; therefore, the laboratory control standard may be prepared in a simpler matrix similar to the calibration verification standard. The Environmental Protection Agency (EPA) or other reference standards could be used for the laboratory control standard; however, these standards normally do not resemble SST matrices or contain all the parameters of interest. For calibration blanks and verification standards, the analysis batch is a set of samples analyzed on the instrument as a unit under identical control conditions or required frequency period. Laboratory control standards and preparation blanks are generated for each preparation batch which is defined as the set of samples prepared as a unit under the same control conditions.

Spikes and duplicates for RCRA and CLP programs are normally based on one in every twenty samples. For SST tanks the frequency has been set to establish an indication of method reproducibility and accuracy for the material in each tank. Therefore, the specification for duplicates and spikes are the same for most parameters. Recently RCRA and CLP have been incorporating the use of matrix spike duplicates (MSD) in the analyses. The advantage of this approach is that precision information is obtained for every spike parameter in the sample matrix even if that parameter may not be present. Duplicate information for parameters at "less than" concentrations are not useful for making precision estimates. The disadvantages of MSDs are that they require an additional preparation if unspiked duplicates also are performed and do not provide actual sample reproducibility if unspiked duplicates are not performed. If only MSDs are used it may be more difficult to evaluate sample homogeneity. The MSDs have been specified only for organic analyses.

There are many ways to establish detection limits and the methods may differ with each parameter. Resource Conservation and Recovery Act (RCRA) detection limit requirements are based on determining the limit in the sample matrix. However, for SST matrices this could be difficult since it is necessary to find samples that do not contain the parameters of interest. This approach may be needed in Phase II to prove that a parameter is below a limit but is not needed in Phase I for general characterization. For stable instruments and analytical systems the detection limit may be determined or verified less frequently than for less stable systems. The detection limit for each parameter should be specified for each segment and core composite data package based on the normal sample size, preparation and analytical method, and operating conditions used. These conditions should be specified for each detection limit provided. The methods for determining detection limits and the frequency of evaluation shall be documented for each applicable parameter.

Acceptance criteria for most parameters are based on SW-846 procedures that considers a value 3 standard deviations (3σ) outside the mean as being out of control. Calibration verification standards may be specified absolutely (90 to 110%) for some parameters rather than by standard deviations or performance. The analyte type may also impact the acceptance criteria since lower priority analytes (Type III) should not require as high a level of performance as Type I and II analytes.

Blanks are used to assess contamination levels in the field, hot cells and laboratory. A system for collecting blank data and setting guidelines for accepting or rejecting data based on the level of contamination in the blank shall be established. A historical mean and three sigma limit shall be determined for the field blank, hot cell blank, preparation (method) blank and calibration blank. The blank results for a parameter shall be compared to its concentration threshold or regulatory limit to determine if the blank result would affect evaluation of sample results at these concentrations. If the blank is less than 10 times these limits or 10 times less than the concentration of the analyte in the sample it is probably insignificant and efforts to remove the contamination source will not be as important. Additional experience and data will be needed in Phase IC to evaluate blank levels and blank control for the high level of inorganic salts found in SST sludges compared to soils and waters.

Duplicate analysis acceptance criteria will depend on the concentration levels in the sample and on the heterogeneity of the sample. It will also depend on the effectiveness of the homogenization method. Both CLP and SW-846 procedures set relative percent difference (RPD) criteria for ICP and AA duplicates at 20% when the analyte concentration is 5 times the contract required detection limit (CRDL) for CLP or 10 times the instrument detection limit for SW-846. The 20% limit is a goal that duplicate analysis of SST waste should attempt to achieve. However, it may not be achievable for these complex wastes that are difficult to homogenize; therefore, the RPD of results should be compared to the historical mean and $\pm 3\sigma$ limits to determine if the performance of the methods have changed significantly for that tank. The method reproducibility also must be evaluated based on the DQO requirements. If confident decisions cannot be made from the data because of the reproducibility, better homogenization and analysis methods will have to be developed. Data whose reproducibility is questionable should be identified and explained. Matrix spike duplicates may be implemented in the future to reduce the effect of low concentration levels.

Matrix spike acceptance criteria also is difficult to establish. Matrix spike recovery criteria for CLP and SW-846, ICP and AA methods are set at between 75 to 125%; however, several factors can keep a sample from meeting this specification. If the spike concentration is only a fraction (25% for CLP) of the sample concentration, variations in the sample concentration will impact the spike recovery. Normally spikes for analytes >0.1 wt% are not evaluated for this reason. Instead, a serial dilution is made that should agree within $\pm 10\%$ of the original analysis. When spikes are added to a separate digestion aliquot the sample heterogeneity becomes a factor in the spike evaluation since the spike aliquot may contain significantly more or less of the constituent than the original aliquot.

The laboratories shall make an effort to determine if a matrix interference exist and identify the cause of the interference. If the spike recovery results are not between 75 and 125%, they should be compared to the mean spike recovery $\pm 3\sigma$ limits to evaluate if this sample represents a significant change in performance. If the results appear to be significantly different than normal, the analytical group should attempt to identify if the problem is one of heterogeneity or a matrix effect on the method by using

techniques such as additional duplicate analyses, or post digestion spikes. Results outside the normal spike performance or post adjustment spikes outside 50 to 150% should be identified and follow-up work and recommendations described. It is important in the early stages of Phase IC to identify matrix interferences and procedure problems so that they may be corrected. The goal of the project is to have methods that provide spike recoveries between 75 and 125% for all analytes; however, this may not be important for the DQOs for all analytes and for all users of the data. The QC performed on SST samples may be restricted by the amount of sample available. The amount of exploratory work to evaluate matrix interferences shall be limited so that schedules and costs to complete the analyses are not significantly impacted.

10.2 SAMPLING QUALITY CONTROL

A minimum of one field blank per tank will be taken. This blank will be used to monitor cleanliness of equipment and transportation effects. An empty sampler will be filled with deionized water in the field and transported to the laboratory where it will be emptied outside the hot cell and analyzed. Initially, the field blank will be analyzed for the following components:

- Total alpha/beta/GEA
- Inductively Coupled Plasma
- Ion Chromatography
- Organics (Gas Chromatography/Mass Spectrometry)
- Total Organic Carbon
- Atomic Absorption (arsenic, selenium, and mercury).

This list may be reduced if experience shows that some of the parameters are not significant. This blank will act as a field, equipment, and trip blank. If significant contamination is found, individual blanks will be prepared to identify the source of contamination. If the sampling for a tank extends over two weeks, a field blank shall be taken near the beginning and near the end of sampling or one every two weeks. If the tank sampling will involve a large number of samples (>20), the field blank frequency shall be 5% of the samplers used. The field blank levels shall be monitored and compared to DQOs, regulatory limits and sample concentration levels. If the blank is significant then the source of contamination should be identified and removed. If the contaminate is a Type III analyte or its concentration threshold limit is large or it is less than 10% of the concentration found in the sample the importance of removing the contaminate will be less.

In Phase IA and IB two cores were taken from the same riser to evaluate the sampling error. This sampling error is made up of errors caused by the sampling technique and variances caused by heterogeneity in the waste. The semisolid character of the waste makes taking two identical samples from the same riser location difficult since taking the first core may disturb the

waste and affect the ability to take the second core. In addition, taking the core from a different riser would provide more useful information on waste heterogeneity and inventory; therefore, this type of duplicate field sample is not planned in Phase IC unless results from Phase IA/IB indicate it may be useful. The program plans to take at least two cores from the same tank but different risers.

10.3 SAMPLE BREAKDOWN QUALITY CONTROL

A minimum of 1 hot cell blank per tank or 1 for every 10 segments extruded shall be prepared. This blank will be prepared by rinsing the precleaned receiving tray and aliquoting equipment with deionized water and collecting it in a normal segment storage container. The storage container with the rinse water is removed from the hot cell and analyzed for the following components:

- Total alpha/beta
- Gamma Energy Analysis
- Inductively Coupled Plasma
- Ion Chromatography
- Total Organic Carbon
- Atomic Absorption (arsenic, selenium, mercury)
- Organics (Gas Chromatography/Mass Spectrometry).

This list may be reduced if experience indicates that no significant quantities of the constituents are found. This blank will monitor the cleanliness of equipment used to receive, store, and subsample the segment samples in the hot cell. Criteria for the hot cell blank is the same as for the field rinse blank.

Extensive sample homogenization tests were performed in Phase IA and IB in which efforts were made to measure individual homogenization and analytical errors. In Phase IC the homogenization and analytical error will be monitored by analyzing at least one segment per core in duplicate for ICP and GEA. Two aliquots will be taken from two extreme (top/bottom or left/right) locations in a homogenized sample. The two aliquots will be analyzed in duplicate using the standard ICP acid digestion and analyzing the final solution for metals by ICP and Cs-137 by GEA. The number of homogenization tests may be increased if segments are found that are more heterogenous than normal or the wastes physical nature is expected to cause homogenization problems. If possible, segments selected for homogenization testing should be chosen so that they represent the different types of wastes in the tank not just wastes with the same consistency.

Reproducibility of compositing segment samples (core composites) will be evaluated on at least one core per tank by preparing two core composites from one set of segment samples. This will monitor how well segment subsamples can be taken, weighted, and homogenized to form a composite. The duplicate core composites will be analyzed in duplicate for the full set of core composite parameters. Acceptance criteria for the homogenization and compositing errors will be based on the historical mean of the relative percent differences (RPD) and its $\pm 3\sigma$ limit.

10.4 METAL CHEMICAL PARAMETERS

10.4.1 Metals--Inductively Coupled Plasma Optical Emission Spectroscopy

The determination of metals by ICP will be used to measure 30 metals of interest. The inductively coupled plasma (ICP) equipment is relatively unstable and it must be calibrated frequently. The calibration requirements in Table D-6 are based on SW-846 and CLP requirements. Calibration verification includes an initial calibration verification followed by calibration checks every 10 samples with an independent verification standard. The calibration verification standard and laboratory control standard can be the same standard providing it is prepared from a different source from the calibration standard and checks all 30 metals. The ICP equipment may also measure another 10 to 20 metals depending on its configuration. However, the QC for these metals does not have to be controlled at the levels described in Table D-6. The instrument should be calibrated for these metals so that if they are present, their concentrations can be estimated. If significant quantities of one of these other metals are found in the tanks, they will be included in the primary analysis set.

The preparation blank monitors the acid, water, and equipment to prepare ICP samples for possible contamination. The blank shall be prepared using a different set of equipment (i.e., beaker, volumetric ware) each time. A preparation blank shall be prepared with each acid digestion, fusion or water preparation batch.

The laboratory control standard (LCS) and continuing calibration verification standard can be the same standard(s). This standard(s) shall contain all the metals of interest. The LCS shall be carried through the same digestion process to evaluate errors associated with digestion and ICP instrument errors. The CLP requires that one duplicate be analyzed from each group of samples of a similar matrix type (soil, water) and concentration (low, high). Proposed RCRA guidelines (EPA 1989) states that the frequency of duplicates shall be based on DQOs for the data collection activity. During the early stages of Phase IC when the DQOs are being developed the frequency of duplicates may be greater than when the DQOs are finally defined. At least two sets of duplicate ICP results should be collected for each tank to permit comparison of errors associated with analysis (homogenization, measurement) and errors caused by tank waste heterogeneity. The objective of the analysis program is to control measurement errors so that they are a minor component of

the overall variability of tank measurements and do not impact DQOs. Because ICP will be used to evaluate homogenization procedures, the ICP duplicates can be performed on segments or core composites. The ICP duplicates should be performed on the acid, water, and fusion digestion aliquots.

The CLP spike frequency requirements are defined the same way as the duplicates described above. The proposed RCRA guidance (EPA 1989) requires a matrix spike with each batch of samples. Batch is defined as a group of samples that behave similarly, have the same matrix chemical properties and are processed as a unit. If the number of samples in a group is greater than 20, then each group of 20 samples or less is handled as a separate batch. The SW-846 ICP method 6010 recommends matrix spike duplicates (MSD) at a 20% frequency. Matrix spike duplicates are not specified for CLP inorganic analyses. Matrix spike duplicates permit both precision and accuracy errors to be determined for the matrix. If the metal concentration is low (less than value), precision estimates from normal duplicates are not possible.

The QC plan in Table D-6 assumes that each core represents a different matrix; therefore a spike is required for each core. When the sample matrix visually changes significantly between segments, additional matrix spikes may be requested if ICP analysis of segments is being performed. The hot cell operations personnel extruding the segments should identify these segments and discuss the option of running additional spikes with the project manager or technical leader. The overall objective is to establish that the method performs properly for the different matrices found in the tank. If a metal is present at >0.1 wt% concentration, spiking is not required; however, matrix effects for these parameters shall be evaluated using a serial dilution. Spikes for ICP analysis of fused samples are not required since this analysis is capable of only measuring metals at relatively high concentrations. Metal spikes should not be added before the water digestion.

Spikes shall be added to samples before the acid digestion. Poor spike recoveries may result from the digestion process, sample heterogeneity, instrument interferences, or too small a spike compared to sample concentration for the parameter. If spike recoveries are outside control limits, a post digestion spike (spike added to a portion of unspiked sample after digestion) shall be used to determine if the problem is associated with digestion, sample heterogeneity or an instrument effect. One additional set of duplicates should be run to further evaluate the sample heterogeneity if it is believed to be the cause of poor spike recovery. If sample heterogeneity is the problem it shall be identified with the data. Decisions on the spike evaluation will depend on the amount of sample available and shall be directed by the technical leader.

Detection limits shall be determined every three months or whenever changes in the instrument could result in a change in performance. Detection limits shall be reported with each data package. The CLP procedure for determining detection limits requires making seven consecutive measurements for the parameters at concentrations 3 to 5 times the estimated instrument detection limit for three nonconsecutive days. New proposed RCRA procedures (EPA 1989) require determining matrix-specific detection limits for

demonstrating compliance with a regulation, DQO or other study objective for any value reported less than a specific regulatory threshold. This procedure requires making three post-digestion matrix spikes additions for the parameters of interest at concentrations of 3 to 5 times the estimated detection limits. This approach also requires finding samples that do not contain any of the parameters of interest. For 20 to 30 parameters, this can be difficult to do; therefore, the CLP approach will be used during Phase IC. The matrix-specific approach may be required in Phase II to verify values reported below regulatory limits.

For matrices such as inorganic sludges, interelement effects can affect the accuracy and detection limits of ICP results. Both CLP and RCRA procedures require interelement correction factors to be checked before and after the end of an analytical run or twice during every 8-hour work shift. The CLP defines the analytes and interferents and their concentration levels for the interference check sample. The SW-846 procedure 6010 is not as well defined and addresses a wider range of analytes and interferents. The SST interelement correction factors should consider all the major metals (>0.1 wt%) found in SST waste as interferents. These will normally include sodium, aluminum, iron, bismuth, and uranium. Uranium is particularly important because of its numerous lines and complex spectra. Some SST waste may contain significant quantities of rare earths which also have complex spectra. If rare earths or other metals are found in the analysis at >0.1 wt% concentrations the interelement corrections for these components must be checked and if necessary added to the interference check sample for that tank. All the metals in the DQO list in Table D-3 shall be evaluated for interference from these major components. When positive results are obtained for a metal not believed to be possible in the SST waste, interference from another waste component should be evaluated.

The CLP procedure uses a contract required detection limit (CRDL) standard (CRA) to verify the linearity of the ICP system near the detection limit. The standard is made up of all analytes except aluminum, barium, calcium, iron, mercury, sodium, and potassium at two times the CRDL or two times the instrument detection limit whichever is the largest. A similar standard shall be used for evaluating SST waste ICP analyses for trace metals. In addition to the metals mentioned above, metals silicon, zirconium, thorium, and uranium do not need to be included. The CLP criteria for these standards is not established; therefore, CRA standards will initially be used for evaluation rather than control purposes.

10.4.2 Metals--Graphite Furnace Atomic Absorption

The GFAA quality control requirements for the SST waste characterization are based on CLP and RCRA (SW-846) procedures. Metals routinely analyzed by GFAA (As, Se, Sb) were identified as Type II or III analytes in the analyte priorities report when they were assumed to be present at 1 wt% concentration in the waste. In actuality their concentration is expected to be at least an order of magnitude less. Therefore, the quality of these results will not be as critical to long-term release risk evaluations as Type I analytes. On the

other hand, these metals are of regulatory interest. Therefore, sufficient quality control is required to provide data with known uncertainty and to evaluate method performance on the waste matrix. Lead (Pb), which is a Type I analyte is occasionally analyzed by GFAA to verify ICP results or to obtain better detection limits.

The calibration requirements for GFAA analyses are essentially the same as for ICP analyses. The single calibration per day is based on CLP procedures. The RCRA (SW-846) procedures recommend hourly calibrations for arsenic and selenium. This was not adopted because long drying and ashing times for some matrices could severely limit the analysis throughput. More frequent calibration verification checks shall be used to identify when recalibration is needed if instrument stability is a problem. The calibration shall include a blank and at least three standards. If the instrument is not designed for these standards in the calibration, calibrate according to the instrument manufacturer's recommendations and use the additional standards to verify the calibration. If the recoveries are outside 90% to 110%, the calibration shall be repeated. A calibration blank shall be run at least every 10 samples to ensure no memory effects are occurring. If memory effects are noted, the condition shall be corrected and the calibration blank frequency increased.

A preparation blank shall be run with each digestion batch. Two sets of duplicate GFAA results (example: duplicate for each of 2 composites) shall be collected for each analyte for each tank unless more sets are specified in the DQOs or test plan. The CLP procedure requires duplicate injections for each sample to evaluate the reproducibility of the injection and furnace system for the matrix. This results in faster graphite tube deterioration and reduces analysis efficiency. For SSTs at least one duplicate injection for every five samples or one per batch shall be done to evaluate instrument reproducibility. If the RPD exceeds 20%, the problem should be corrected and/or the frequency of duplicate injections increased.

The GFAA analysis by CLP procedures requires the use of both predigestion and post digestion spikes and a complicated scheme of spike evaluation. This level of control is not considered necessary for Phase I analysis of Type II analytes. In the SST characterization program at least one predigested spike should be performed on each core or with significant matrix changes within the tank. The hot cell operations group shall have the lead in identifying when significant matrix changes occur and additional spikes are needed. Each sample (except the predigested spike sample) shall be analyzed with a post digestion spike. If the spike recovery results are not between 50% and 150%, the data shall be flagged and the problem documented. If appropriate the method of standard additions should be used to evaluate samples with spike recoveries outside these limits.

The method of standard additions (MSA) may provide more accurate analyses for some types of matrix interferences; however, it is not effective for spectral interferences. The technical leader is responsible for deciding if MSA should be used depending on such factors as the potential source of error, the magnitude of the error and the concentration level and priority of the analyte.

The CLP procedures use contract required detection limit (CRDL) standards to evaluate the instrument performance near the detection limit. Since GFAA metals are expected to be low or near detection limits, a CRDL standard shall be analyzed twice in an 8-hour operation period. Detection limits shall be determined every 3 months and reported with all data during that period.

10.4.3 Metals--Hydride Atomic Absorption

Hydride atomic absorption analyses also may be used to analyze for arsenic, selenium, and antimony. The QC requirements for HYAA are based on SW-846 procedures. The HYAA system shall either be calibrated hourly using a blank and three standards or data should be bracketed with calibration verification standards. A calibration verification or laboratory control standard and a preparation blank shall be run with each batch of samples. If spike recoveries routinely fall outside the 50% to 150% range, an attempt to identify the cause of the error shall be made by evaluation of sample heterogeneity or if appropriate the method of standard additions (MSA) applied. Detection limits shall be determined and reported on a quarterly basis.

10.4.4 Metals--Cold Vapor Atomic Absorption

Mercury is the only metal analyzed by cold vapor atomic absorption (CVAA) techniques. The QC is based on SW-846 procedures which requires hourly calibrations with a blank and three standards. The CLP requires a daily calibration frequency. If hourly calibration impacts analysis efficiency, daily calibration can be used provided the data is bracketed with calibration verification standards in the same range as the sample. If the calibration verification standard recoveries are outside of 80% to 120%, the instrument must be recalibrated and the samples reanalyzed. The method of standard additions (if appropriate), method changes, or heterogeneity effects shall be evaluated if spike recoveries are outside 50% to 150%. Detection limits shall be determined and reported on a quarterly basis.

10.4.5 Metals--Fluorimeter

Total uranium is determined using a laser fluorimeter technique based on a single standard addition; therefore, the instrument is calibrated with each sample. At least one calibration blank shall be run with each batch of samples analyzed on the fluorimeter to ensure cells are being properly cleaned. If the blank is greater than 5% of the sample concentration level, the previous samples shall be reanalyzed. Preparation blanks are used to check for contamination in the fusion procedure. The method detection limit shall be defined and the basis for determining the limit documented. The detection limit shall be reestablished anytime the equipment or procedure changes occur that may impact the method performance.

10.4.6 Metals--Ultraviolet Spectrophotometry

Chromium VI may be determined by using spectrophotometry. This equipment and method are more stable than ICP and AA techniques and do not require as frequent calibrations. This approach is supported in new guidelines discussed by Environmental Protection Agency personnel (Friedman 1990). Calibrations shall be checked after equipment maintenance or reagent changes. A reagent blank is run with each batch of samples. The calibration shall be checked with each batch of samples using either an independent calibration verification standard or a laboratory control standard. The method detection limit shall be defined and the basis for determining the limits documented. The detection limits shall be reestablished any time equipment or procedure changes occur that may impact the methods performance.

10.5 ANION CHEMICAL PARAMETERS

10.5.1 Anions-Ion Chromatography

The QC requirements for ion chromatography are based on "The Environmental Survey Manual" (DOE 1987) and EPA Test Method 300.0-1 (EPA 1989). After initial calibration, the calibration verification standard or laboratory control standard shall be used before and after a set of analyses and at least after every 10 samples. The instrument must be recalibrated when the eluent is changed or when the calibration or control standard recoveries are outside the 90% to 110% limits. A calibration blank should be analyzed after every 10 samples to ensure the columns are being adequately flushed between samples. The retention time shall be checked with each calibration verification. If the retention time has shifted by more than 10% from the expected value, the run shall be stopped and the cause of the shift identified and a new calibration prepared if required. The detection limits for each anion shall be defined and the basis for determining the limits documented. The detection limits shall be reestablished any time equipment or procedure changes occur that may impact the methods performance.

10.5.2 Anions--Spectrophotometry

Nitrite is determined by spectrophotometry in the 222-S facility with QC requirements essentially the same as those for Cr(VI) under metals. Because nitrite is easily oxidized by air, standards shall be prepared on a frequent basis (at least weekly).

10.5.3 Anions--Selective Ion Electrode

The PNL 325 Laboratory personnel determine fluoride by selective ion electrode (SIE). The instrument is calibrated before analysis of each batch of samples. Calibration may be done graphically using at least three standards or direct concentration measurements using two standards. Analysis of samples must be bracketed by the standards. The calibration shall be

checked using the calibration verification standard or the laboratory control standard once every 10 samples and after each batch. The detection limits for SIE shall be established with each new electrode and any procedure change that may affect its response.

10.5.6 Anions--Distillation

Cyanide and sulfide are determined by distillation and final measurement using spectrophotometry, titration, or selective ion electrode. The distillation of these anions requires a long period of time and limits the throughput of analyses; therefore, they are performed with reduced QC for some areas. The calibration requirements for cyanide are essentially the same as for Cr(VI) and nitrite. The calibration blank is a reagent blank for the spectrophotometer. A preparation blank is a check on the distillation equipment and reagents used for distillation. One of these shall be prepared at the end of each day (8 hours). The preparation blank shall be prepared using a different distillation system each time so that all systems are continually checked for contamination. The verification standard is a standard that is not carried through the distillation but is analyzed with each batch of spectrophotometric samples. The laboratory control standard is carried through the distillation to verify its operational efficiency. At least one laboratory control standard shall be run every 8 hours using different distillation systems in the same manner as the preparation blank. If a microdistillation system that can distill multiple (10 or more) samples is used, a preparation blank and laboratory control standards shall be analyzed with each distillation batch.

10.5.7 Anions--pH

The determination of pH is calibrated each time using two standard buffers before making measurements. The pH calibration shall be verified every 10 samples or after each batch of analyses to ensure the electrode has not drifted. If the pH of the standard has drifted by more than ± 0.05, the electrode shall be recalibrated and the samples rerun. Calibration and preparation blanks are not required since the pH of the samples is not expected to be affected by the pH of the deionized water used to prepare the sample. Matrix spikes and detection limits are not applicable.

10.5.8 Anions--Total Inorganic Carbon

Carbonates are determined using TIC/TOC equipment. The carbon dioxide gas release is either measured by nondispersive infrared (NIR) analyzer or by coulometric titration. Since calibration procedures vary with each type of instrument, the calibration shall be performed according to the instrument manufacturer. The NIR systems normally are calibrated with several standards before each batch of analyses with verification performed during analysis of samples (1 per 10 samples) and at the end of the run. Verification standard recoveries shall be within 90% to 110% or the instrument recalibrated and samples rerun. The coulometric titration systems require only a verification

standard run before, during (1 per 10 samples), and after analyses since coulometry is a primary standardization technique. The method detection limit shall be defined and its basis documented. The detection limit shall be reestablished any time equipment and procedure changes occur that impact the methods performance.

10.5.9 Other Methods--NH_3, $Fe(CN)_6^{-4}$

Ammonia is determined by distillation and titration at the 222-S Laboratory. No calibration or calibration blank is necessary but a laboratory control standard shall be used to verify the distillation equipment and titration reagents produce acceptable results. At least one laboratory control standard and preparation blank (distillation blank) shall be prepared every 8 hours like the cyanide systems. The PNL 325 Laboratory uses SIE to determine ammonia without the need for distillation. The QC requirements for this procedure are the same as those for fluoride.

The method for ferrocyanide speciation is still under development, therefore QC requirements for the instruments and procedure cannot be established.

10.6 ORGANIC PARAMETERS

10.6.1 Organic--Total Organic Carbon (TOC)

The TOC QC requirements are essentially the same as those for carbonate or TIC.

10.6.2 Organic--Total/Extractable Organic Halides (TOX/EOX)

The coulometric chloride analyzer calibration on the TOX/EOX system shall be checked daily using a chloride standard. If the calibration does not agree within ±2% the instrument parameters shall be adjusted according to the instrument manufacturer. The performance of the entire TOX/EOX system shall be checked by triplicate injections of an independent check standard after every 10 samples. The standard recovery should be within 80 to 120% and the standard deviation of the injections shall be $\leq \pm 10\%$. A preparation blank (EOX extractant) and laboratory control standard shall be analyzed with each batch of samples. The method detection limit shall be defined and its basis documented. The detection limit shall be reestablished any time equipment or procedure changes occur that may impact the methods performance.

10.6.3 Organic--Complexants

The method for determining organic complexants has not been determined; therefore, QC requirements for the instrument and procedure cannot be established.

10.6.4 Organic--GC/MS (Volatile Organics by Purge and Trap and Semivolatile Organics by Capillary Columns)

The volatile organic analysis QC requirements are based on the most recent CLP Statement of Work (SOW) (EPA 1990). The EPA is in the process of trying to develop one set of methods and QC requirements for both CLP and RCRA programs. Since the CLP SOW represents the most recent change in procedures, it is assumed that it is the direction the EPA is going. The QC requirements for GC/MS analysis of volatiles and semivolatiles are very detailed and will not be provided in this document. For details the CLP SOW and applicable RCRA (8240, 8270) procedures should be consulted. Table D-6 and this discussion provide general guidelines for the QC of these methods. Calibrations must be checked every 12 hours with a one point standard. Before calibrations are performed, the GC/MS must be tuned according to the CLP procedure. Before analyzing samples the system performance check compounds (SPCCs) and calibration check compounds (CCCs) must be run and meet CLP performance criteria. The CCCs and SPCCs are also included in the initial calibration. These same check compounds and preparation blank must be analyzed every 12 hours. Each blank, standard, and sample are analyzed with a surrogate standard and an internal standard. Since the CLP and RCRA procedures require at least one matrix spike and one matrix spike duplicate for every 20 samples, only one set (rather than two) has been specified per tank. Duplicates (without spikes) are not required by CLP and RCRA GC/MS procedures; however, one duplicate per tank should be performed to evaluate sample heterogeneity. Spiked duplicates may overshadow sample heterogeneity effects.

There appears to be two major QC differences in CLP and RCRA procedures. Since each sample is spiked with known surrogates and internal standards, the CLP procedure does not require a laboratory control standard. The RCRA procedure requires a quality control check standard (laboratory control standard) with each batch. The other area, which is different, is the acceptance criteria for these control checks. The CLP procedure has specified the acceptance criteria, whereas RCRA allows the lab to establish its own criteria based on the actual variances observed. The goal of the analysis shall be to meet the CLP criteria; however, the laboratories shall collect statistical information to set 3σ limits for the standard, duplicate, and spike measurements to evaluate the method performance on SST matrices and for comparison to CLP and SW-846 performance specifications. The varying NPH contamination and matrix uncertainties are expected to produce problems with surrogate and matrix spike recoveries. If larger samples (10g) are needed to meet the required DQOs, such as TCLP compound limits and Ecology guidelines of 10μg/g for organics, there may be insufficient sample for all QC tests. One set of duplicates and matrix spike duplicates require at least 40g of sample for each type of organic analysis. If inadequate sample is available, the duplicate and then the matrix spike duplicate shall be dropped from analysis, in that order, depending on sample quantities available.

The primary objective of these organic analyses is to prove that significant quantities of organics are present in the wastes and if any of the organics are of regulatory interest, or could contribute to the overall risks in the tank. Since the objective is not to prove that the organics are below

regulatory levels, the level of QC for the analyses in Phase I does not need to be as high as in Phase II. Laboratories shall make reasonable attempts to meet CLP requirements and document the results when these requirements cannot be met.

10.7 Radiochemical Parameters

10.7.1 Radionuclides--Alpha

The QC requirements for radionuclides is based on the EPA Handbook for Analytical Quality Control in Radioanalytical Laboratories (EPA 1977). The alpha counters used in the Westinghouse Hanford and PNL laboratories are either window or windowless gas-flow proportional counters or zinc sulfide scintillation counters. These detection systems normally are stable after initial calibration and only need to be checked to ensure they are operating properly. All radiochemical calibrations shall be done with standards traceable to National Institute of Standards and Technology (NIST). Calibrations shall be checked following any maintenance activities that could modify equipment performance.

If alpha energy analysis (AEA) systems are used for determining absolute alpha concentrations (not just ratios) the same calibration conditions apply. In addition, the alpha energy peak locations shall be checked and adjusted as required by the operating procedure, unless the equipment performs a self calibration of alpha energies with each measurement. Backgrounds must be acquired for each detector system on a regular basis. The recommended frequency for taking the background depends on the normal counting time used for analysis of samples as noted below:

Count Time	Background Frequency
0 - 1 hour	1 per 8 hours
1 - 8 hours	1 per 24 hours
>8 hours	1 per week

These frequencies may vary depending on if the counting room operates on a single 8 hr shift or three 8 hour shifts. At least one background shall be determined per day unless count times exceed 8 hour. These same conditions apply to check standard frequency.

If the background count is outside the $\mu \pm 3\sigma$ control limit, recount to confirm it is out of control and clean the detector. Samples between the last good background and out of control background shall be recounted using the new background level. If the count rate for the samples are high enough that the change in background will not significantly (+20%) affect the result, recounting the samples is not necessary. Example: The background shifts from 0.1 to 1.0 c/m but the sample count rate is 100 c/m, recounting is not required. However, if the sample count is 5 c/m or less, it shall be recounted using a cleaned detector. Alpha energy systems normally require longer count times because of their lower efficiency; therefore, backgrounds shall be taken at least once per week. The calibration of the alpha system

shall be verified with a check standard at least once every 24 hours when the equipment is in operation. The check standard count rate is normally high enough that long count times are not required. Also it should be high enough concentration to have counting errors at <5%. If the check standard is outside its 3σ limit, it shall be recounted to verify the out-of-control condition. If it is confirmed that the system is out of control, it shall be recalibrated and the prior samples recounted.

A preparation blank is prepared for each digestion (fusion) batch and processed with the samples to evaluate contamination in the analytical system. If contamination is found that is significantly (μ+3σ) over the background, the cause of the contamination shall be identified and removed. Most radiochemical analyses are performed with disposable equipment to eliminate the potential for contamination.

A laboratory control standard is processed with each batch of samples. If adequate standard isotope quantities are available, a mixed isotopic laboratory control standard shall be prepared at concentrations high enough to permit accurate (±2 to 5%) counting rates after fusion, digestion separation, and mounting. If there is insufficient isotopic standards, the laboratory control standard is prepared at a lower concentration and carried only through the separation method. If the laboratory control standard is outside the 3σ control limit, the out-of-control condition shall be verified, the reason identified and corrected, and samples in the batch reanalyzed. If the condition cannot be corrected the data should be flagged and the standard recovery noted.

Matrix spikes are not required for radiochemical analyses if an isotopic tracer is used in the analysis. Since these tracers are becoming difficult to obtain they must be used in limited quantities. Therefore, they are used only to determine the yield for the chemical separation portion of the procedures. ^{242}Pu or ^{236}Pu are used as tracers for plutonium. Americium-243 is used as a tracer for Am-241 and curium isotopes. The tracers for Pu and Am can be distinguished from the isotope being analyzed by AEA and therefore do not require a separate aliquot for analysis. Neptunium-239, a gamma emitter, is used as a tracer for ^{237}Np. It requires counting on both alpha and gamma systems and because of the short half-life of ^{239}Np the results must be decay corrected for each analysis. The laboratories shall report the percent recovery for each isotopic tracer. If an isotopic tracer is not available or not used, a matrix spike on a separate sample aliquot using the same isotope that is being analyzed must be used. The matrix spike shall be at a concentration of at least 5 to 10 times the sample isotope concentration so that sample reproducibility effects are minimized. Isotopic tracers must be standardized and maintained as high quality standards since any variation in the tracer will be reflected in the final results. The goal of the program requires procedures with tracer recoveries >50%. To evaluate spike recoveries, historical spike data will be maintained and development tasks will be identified for procedures with <50% yields. A matrix spike for total alpha should be used to evaluate solids absorption effects on the results. Spikes should be made at least 5 to 10 times the sample concentration. If the absorption effects of the solids exceeds 20%, the samples will be diluted (if possible) and reanalyzed.

Detection limits for counting methods depend on many variables. Detection limits for the radiochemical analysis on SST waste shall be reported based on the normal sample size, preparation method, separation method, count time, detector efficiency, and background. These conditions shall be specified for each isotope detection limit. These must be reported with each data package or whenever conditions, methods, or detection limits change significantly (+25%). In addition to detection limits the 2σ counting error shall be reported for each radioisotope and each analysis. This provides information on the minimum reproducibility error for the analysis and indicates how confident the data can be used.

A plateau and operating voltage for alpha counters shall be determined at least every 12 months. The sensitivity of the alpha counters to counting betas is important for SST analyses since the ratio of beta activity to alpha activity is normally very large. The sensitivity of alpha counters to beta shall be determined by counting a high activity beta source on alpha counting equipment at least every month. If the beta counts are significant (could result in reporting false TRU >100nCi/g levels), the operating voltage shall be adjusted accordingly or the detector shall be limited to counting samples with low beta activity.

10.7.2 Radionuclides--Beta

Radionuclides--Beta. The QC for determining beta emitting isotopes is essentially the same as for alpha isotopes. The major differences are in the use of matrix spikes and the evaluation of beta purity. Rather than isotopic tracers most beta isotope methods use non-radioactive carriers of the same element to determine the chemical yield through the separation procedure. Carriers are added only for the separation method not at the digestion (fusion) stage. The carrier recoveries for the isotope shall be reported for each analysis. The ^{85}Sr tracer may be used for ^{90}Sr analysis; however, present procedures use a Sr carrier. ^{63}Ni, ^{129}I, and ^{79}Se all use carriers of the respective elements. The ^{95}Tc isotope may be used as a tracer for ^{99}Tc; however, since it may not be available and since there are no non-radioactive carriers, a matrix spike of ^{99}Tc at concentrations at least 5 to 10 times the sample concentration shall be used. Total beta results shall be reported based on ^{90}SrY beta efficiency. No matrix spike is required for total beta results since absorption effects are less than alpha, large dilutions are normally required, and the total beta results are used only for an indication of total activity.

Analysis of minor (^{129}I, ^{79}Se, ^{99}Tc, ^{63}Ni) isotopes in SST waste represents one of the most difficult problems because of the high levels of ^{90}SrY and ^{137}Cs, which can potentially interfere in the other analyses, if efficient isotopic separations are not obtained. The beta purity of the separation for each procedure should be established on SST matrices to show that no other beta isotope is contributing to the result. This is particularly important for isotopes that exceed the threshold limits. Beta purity may be verified by multiple absorber techniques in which a sample containing adequate beta counts is counted repeatedly with increasing density absorbers. Gamma energy analysis (GEA) analyses of the beta mount also can be used to indicate the

presence of other isotopes such as ^{137}Cs. Beta purity measurements are not required for each determination but should be performed on a few samples from tanks in each operable unit. Once it is documented that the procedures being used have no other beta interferences beta purity measurements can be eliminated.

10.7.3 Radionuclides--Beta/Liquid Scintillation

Beta isotopes also may be determined by liquid scintillation counting. Tritium (H-3) and carbon-14 (C-14) are the most common isotopes analyzed by liquid scintillation. The technology for liquid scintillation varies significantly between manufacturers; therefore, calibration procedures shall be based on manufacturer recommendations. Liquid scintillation counters normally use automatic sample changers that count a background and verification standards with each batch of samples. The preparation blank evaluates contamination from the analysis steps and shall be prepared with each batch of samples digested. Carbon-14 and H-3 matrix spikes shall be added to the distillation steps to estimate the efficiency for distillation. Carriers may be used to evaluate the yield for other isotopes analyzed by liquid scintillation as described for beta counting methods. Liquid scintillation systems shall provide for quench correction of the results (when applicable) based on manufacturer recommendations or operating instructions. Beta purity on some liquid scintillation systems can be evaluated by printing out a beta spectrum. This shall be done whenever SST samples with different isotopic compositions or from different operable units are analyzed. The checking of the liquid scintillation samples by GEA may be used to verify purity.

10.7.4 Radionuclides--Gamma Energy Analysis

Gamma energy analysis systems must be calibrated for each geometry used and over the gamma energy range of all the isotopes analyzed. The GEA instruments are normally stable and do not require recalibration unless the check standard indicates the system is out of control. The energy efficiency curve shall be determined annually for each detector and geometry with a multienergy reference standard(s) traceable to NIST standards. The detector background shall be checked at least every 24 hours when the system is in operation. Less frequent backgrounds may be taken for longer count times as discussed in the alpha section. A check standard with multiple gamma energies over the range of interest shall be run at least once every 24 hours during operation. The check standard needs to be evaluated for efficiency (quantitative recovery) and for energy linearity. The check standard must include peaks in the low energy region where linearity is the most critical. The check standard shall be used to make any adjustments necessary in energy peak locations to normalize the instrument to calibration conditions. If the check standard recovery or linearity is outside the 3σ limits, the system must be evaluated further and recalibrated if necessary. Samples shall not be analyzed on a system that is out of control. All samples analyzed on an out of control system will be reanalyzed.

The laboratory control standard shall be processed through the digestion, dilution, and mounting steps if sufficient standard quantities are available. If sufficient standards are not available, the control standard shall be processed through only the dilution, mounting, and analysis steps. No matrix spike is needed for GEA analysis since no separation steps are involved and absorption effects are not significant. Low energy photon spectroscopy (LEPS) gamma detection systems are used for analysis of low energy (X-ray) emitting isotopes such as ^{129}I. For ^{129}I cold iodine carrier or a matrix spike shall be used to determine separation yield and evaluate matrix effects. Carrier or spike recoveries shall be reported for each analysis. Detector resolution should be checked and documented at least once per month.

10.7.5 Radionuclides--Mass Spectrometry

Uranium and plutonium isotopes are determined by thermal ionization mass spectrometry. This technique is used to establish the relative concentrations of each isotope (isotopic ratios) which is then used with alpha Pu information to determine ^{240}Pu and ^{241}Pu isotopic concentrations and with total U by laser fluorometry to determine ^{234}U, ^{235}U and ^{238}U isotopic concentrations. A New Brunswick Laboratory (NBL) plutonium and uranium isotopic standard is analyzed with each batch of samples to calibrate and verify the mass spectrometer performance. A preparation blank is analyzed with each batch to check for contamination in the system. A matrix spike is not required since only isotopic ratios are being measured. A detection limit shall be specified for the normal sample size, separation and instrument. This limit is used only to evaluate if plutonium and uranium concentrations are high enough to allow isotopes to be determined on the waste and, therefore, only needs to be reported when it changes significantly.

Inductively coupled plasma mass spectrometry (ICP-MS) may be used to determine long-lived isotopes such as actinides, ^{99}Tc and ^{129}I. Procedures for this new technology are in the process of being developed and should address the major quality control areas outlined in Table D-6. Environmental Protection Agency methods for ICP-MS are expected in the future and shall be used as guidance for QC procedures for SST waste analyses.

10.8 PHYSICAL MEASUREMENTS PARAMETERS

10.8.1 Physical Measurements--Bulk Density

This measurement has no calibration requirements. Balances used in the measurement shall be calibrated at least annually. If the balance has an internal check weight, it shall be checked before each batch of weighings. Volumetric equipment must be accurate to ±2%. Duplicate data is gathered to evaluate the variability in the test unless it is determined from weight and dimensions or there is insufficient sample. Other QC areas such as matrix spikes and detection limits are not applicable.

10.8.2 Physical Measurements--Particle Size

Equipment shall be calibrated according to the instrument manufacturer. A laboratory control standard or calibration verification standard shall be run with each batch. Blanks are not required for SST samples.

10.8.3 Physical Measurements--Penetrometer

The manual mechanical penetrometer is a crude device used to determine if the waste is dilatant or cohesive; therefore, the QC requirements are minimal. Two reading per segment shall be taken to evaluate the reproducibility of the system unless it is obvious the waste is so soft that only less than readings will be obtained or sample size and configuration permits only one reading. The penetrometer mechanical condition shall be evaluated visually to ensure that corrosion of the spring or piston have not become excessive. If the system is showing signs of deterioration, the penetrometer shall be replaced.

10.8.4 Physical Measurements--Viscosity and Rheology

Rheology systems shall be calibrated in accordance with manufacturer recommended procedures. Blanks are not applicable to rheology measurements. A calibration verification standard shall be run with each batch of samples or at a frequency recommended by the manufacturer. No laboratory control standards simulating waste rheology are available. No standards have been identified for shear strength measurements. Duplicate samples shall be run if sufficient samples are available. Replicate rheology readings shall be taken for a single sample. Present rheology methods use 50-100g of sample, which may limit the number of duplicates. Duplicates shall not be run if sample is needed for additional tests.

10.8.5 Physical Measurements--Porosity and Compressive Strength

These measurements are intended only for hard saltcakes and sludges. Methods must be developed before the QC requirements can be defined.

10.8.6 Physical Measurement--DSC/TGA

The DSC/TGA equipment shall be calibrated according to the manufacturers recommendations or once per quarter. The equipments calibration for temperature, heat and weight measurement should be checked with each batch of samples. Any sample exhibiting an exotherm shall be analyzed in at least duplicate to verify the results.

10.8.7 Physical Measurement--wt% Water

The weight percent water shall be determined in duplicate for each sample immediately prior to subsampling and analysis of each sample (segment or core

composite). The weight percent water analysis shall be repeated before any other analysis is performed on a sample stored for 30 d or more. A laboratory control standard shall be analyzed with each batch of samples.

10.8.8 Physical Measurement--Other

Several physical measurements have been identified as part of the necessary characterization information for tanks involving Unresolved Safety Questions. The measurements needed for the high heat tanks are thermal output, coefficient of thermal expansion, specific heat, thermal conductivity and thermal conductivity of the frozen sludge. In gas generating tanks, a measurement determining deliquescence has been identified as being necessary characterization information.

10.9 CHARACTERISTICS PARAMETERS

10.9.1 TCLP

A blank extraction is prepared with each batch of samples from a tank. The ICP and AA analyses are performed using the same guidelines as the normal procedures except spikes are added only to the TCLP extract and not the original sample before TCLP extraction.

10.9.2 Chemical Oxygen Demand

The method for determining chemical oxygen demand has not yet been developed; therefore, QC requirements for the method cannot be established.

Table D-6. Quality Control Guidelines for Characterization of Single-Shell Tank Wastes. (sheet 1 of 6)

Parameter	Calibration	Calibration blank[a]	Calibration verification	Preparation blank[a]	Laboratory control standard	Duplicates	Matrix spikes	Detection limits	Other	Requirement
Sampling	NA	NA	NA	1 per tank or 5% criteria: $\leq \mu \pm 3\sigma$	NA	2 cores per tank	NA	NA	NA	NA
Sample break down	NA	NA	NA	1 per tank or 10% criteria: $\leq \mu \pm 3\sigma$	NA	NA	NA	NA	NA	NA
Homogenization	NA	NA	NA	NA	NA	1 seg/core criteria: RPD $\leq \mu \pm 3\sigma$	NA	NA	NA	NA
Compositing	NA	NA	NA	NA	NA	1cc/tank criteria: RPD $\leq \mu \pm 3\sigma$	NA	NA	NA	NA
Analysis										
Metals--ICP 30 metals	1/24 h or each setup	1/ea 10 samples or 1/batch after each calibration, verification and batch Criteria $\leq \mu \pm 3$	1/ea 10 samples or 1/batch Criteria: Type I-II 90 to 110% Type III $\pm 3\sigma$	1/digestion batch Criteria: $\leq \mu \pm 3\sigma$	1/batch Criteria: $\mu \pm 3\sigma$ % Recovery	2 sets of duplicate result per tank or as required by DQOs Criteria: RPD <20% or RPD $\leq \mu \pm 3\sigma$	1 spike/core or with matrix change Criteria Type I and II 75 to 125% or recovery $\mu \pm 3\sigma$	Measured 1/3 mo	Interelement correction Interference check sample CRDL std. Serial dilution	Measure 1/3 mo 2/8 h Criteria ±20% 2/8 h 1/core Criteria: 10% of original
Metals--GFAA As, Se, Sb	1/24 h or each setup	1/ea 10 samples or 1/batch Criteria: $\leq \mu \pm 3\sigma$	1/ea 10 samples or 1/batch Criteria: Type I-II 80 to 120% Type III $\pm 3\sigma$	1/digestion batch Criteria: $\leq \mu \pm 3\sigma$	1/batch Criteria: $\mu \pm 3\sigma$ % Recovery	2 sets of duplicate result per tank or as required by DQOs Criteria: RPD <20% or RPD $\leq \mu \pm 3\sigma$	1 prodigest spike per core or with matrix change 1 postdigestion spike per sample Criteria: Type I and II 75 to 125% or recovery $\mu \pm 3\sigma$%	Measured 1/3 mo	MSA (If appropriate) CRDL std Duplicate injections	Post spike recovery <50% >150% 2/8 h 1/5 samples or 1/batch Criteria: RPD <20%
Metals--HYAA As, Se[b]	Once/h or bracketed data with calibration verification	Each calibration or 1/batch Criteria: $\leq \mu \pm 3\sigma$	1/every 10 samples or 1/batch Criteria: 90 to 110%	1/batch Criteria: $\leq \mu \pm 3\sigma$	1/batch Criteria: $\mu \pm 3\sigma$ % Recovery	2 sets of duplicates per tank or as required by DQOs criteria	1 spike per core per tank or with matrix change criteria	Measured 1/3 mo	MSA (If appropriate)	Spike recovery <50 or >150%
Metal--CVAA Hg[b]	Once/h or bracketed data with calibration verification	1/batch Criteria: $\leq \mu \pm 3\sigma$	1/each 10 samples or 1/batch Criteria: 80 to 120%	1/batch Criteria: $\leq \mu \pm 3\sigma$	1/batch Criteria: $\mu \pm 3\sigma$ % Recovery	2 sets of duplicates per tank or as required by DQOs	1 spike per core per tank or with matrix change	Measured 1/3 mo	MSA (If appropriate)	Spike recovery <50 or >150%

Table D-6. Quality Control Guidelines for Characterization of Single-Shell Tank Wastes. (sheet 2 of 6)

Parameter	Calibration	Calibration blank[a]	Calibration verification	Preparation blank[a]	Laboratory control standard	Duplicates	Matrix spikes	Detection limits	Other	Requirement
Metals--fluorimeter U[b]	Method standard addition	1/batch Criteria: $\leq \pm 3\sigma$	Each sample Criteria: NA	1/digestion batch Criteria: $\leq \pm 3\sigma$	1/batch Criteria: $\pm 3\sigma$ % Recovery	2 sets of duplicates per tank or as required by DQOs	1 spike per core per tank or with matrix change	DQR	NA	NA
Metals--Spec Cr(VI)[b]	1/yr or whenever LCS or calibration verification % recovery is outside 90 to 110%	1/10 samples or 1/batch (reagent blank) Criteria: $\leq \pm 3\sigma$	1/batch Criteria: 90 to 110%	1/digestion batch Criteria $\leq \pm 3\sigma$	1/batch Criteria: $\pm 3\sigma$ % Recovery	2 sets of duplicates per tank or as required by DQOs	1 spike per core per tank or with matrix change	DQR	NA	NA
Anions--IC NO_3^-, NO_2^-, F^-, Cl^-, SO_4^{-2}, PO_4^{-3}[b,c]	Initial or when event changed or when verification standard is out of control	Each calibration or 1/10 samples	1/10 samples before and after each calibration or 1/10 samples Criteria: 90 to 110%	1/batch	1/batch	2 sets of duplicates per tank or as required by DQOs	1 spike per core per tank or with matrix change	DQR	Retention time	If changed >10% recalibration
Anions--UV spec NO_2^-[b,c]	1/yr or whenever LCS or calibration verification % recovery is outside 90 to 110%	1/batch	1/batch Criteria: 90 to 110%	1/batch	1/batch	2 sets of duplicates per tank or as required by DQOs	1 spike per core per tank or with matrix change	DQR	NA	NA
Anions--SIE F^-[b,c]	Method standard addition or each batch	1/batch	Each sample or after each batch and 1/10 samples criteria: 80 to 120%	1/batch	1/batch	2 sets of duplicates per tank or as required by DQOs	1 spike per core per tank or with matrix change	DQR	NA	NA
Anions--UV spec CN^-, S^{-2}, (distillation)[b,c]	1/yr or whenever LCS or calibration verification % recovery is outside 90 to 110%	1/batch (no distillation)	1/batch (no distillation) Criteria: 90 to 110%	1 distillation blank every 8 h	1/batch or every 8 h	2 sets of duplicates per tank or as required by DQOs Criteria	1 spike per core per tank or with matrix change criteria.	DQR	NA	NA
Anions--pH, OH^-[b,c]	Each batch	NA	End of each batch and 1/10 samples Criteria: ±0.05 pH	NA	1/batch	2 sets of duplicates per tank or as required by DQOs	NA	NA	NA	NA

Table D-6. Quality Control Guidelines for Characterization of Single-Shell Tank Wastes. (sheet 3 of 6)

Parameter	Calibration	Calibration blank[a]	Calibration verification	Preparation blank[a]	Laboratory control standard	Duplicates	Matrix spikes	Detection limits	Other	Requirement
Anions--TIC, CO_3^{2-}[b,c]	APIN	Each calibration and 1/batch and 1/10 samples	1/batch or 1 every 10 samples and/or after each batch Criteria: 90 to 110%	1/batch	1/batch	2 sets of duplicates per tank or as required by DQOs	1 spike per core per tank or with matrix change	DQR	NA	NA
Other--SIE NH_3	Method standard addition or each batch	1/batch	Each sample or after each batch and 1/10 samples Criteria: 80 to 120%	1/batch	1/batch	2 sets of duplicates per tank or as required by DQOs	1 spike per core per tank or with matrix change	DQR	NA	NA
Other--Titration NH_3[b,c]	NA	NA	NA	1 distillation blank every 8 h or 1/batch	1/batch or every 8 h	2 sets of duplicates per tank or as required by DQOs	1 spike per core per tank or with matrix change	DQR	NA	NA
Organic--TOC	APIN	Each calibration and 1/batch or after every 10 samples	1/batch or 1 every 10 samples and/or after each batch Criteria: 90 to 110%	1/batch	1/batch	2 sets of duplicates per tank or as required by DQOs	1 spike per core per tank or with matrix change	DQR	NA	NA
Organic--EOX/TOX halogenated carbon[b,c]	Daily criteria: ±2%	Each calibration and 1/batch or 1 every 10 samples	Before and after each batch of samples and 1 every 10 samples	1/batch	1/batch	2 sets of duplicates per tank or as required by DQOs	1 spike per tank or with matrix change	DQR	NA	NA
Organic--TBD complexants	TBD	TBD	TBD	TBD	TBD	2 sets of duplicates per tank or as required by DQOs	1 spike per core or with matrix change	TBD	NA	NA

Table D-6. Quality Control Guidelines for Characterization of Single-Shell Tank Wastes. (sheet 4 of 6)

Parameter	Calibration	Calibration blank[a]	Calibration verification	Preparation blank[a]	Laboratory control standard	Duplicates	Matrix spikes	Detection limits	Other	Requirement
Organics--GC/MS volatile	Initial and 1/12 h Initial SPCCs, CCCs	MA	1/12 h SPCCs, CCCs Criteria SPCC: RRF >0.3 Criteria: CCCs: ≤±25% of initial	1/12 h or per batch Criteria: ≤±3σ or <CRDL with exceptions	NR	1 duplicate per tank or as required by DQOs Criteria: ±3σ	1 set of MS and MSD per tank or as required by DQOs Criteria: ±2σ	DQR	GC/MS-tune Surrogate std Internal std	1/12 h Each calibration std, each blank, each sample MS and MSD Each calibration std, each blank, each sample MS and MSD Criteria: ±3σ or outside CLP limits
Organics--GC/MS semivolatile	Initial and 1/12 h Initial SPCC and CCCs	MA	1/12 h SPCCs, CCCs Criteria SPCC: RRF >0.05 Criteria CCCs ≤±25% of initial	1/20 samples or per batch Criteria: ±3σ or <CRDL with exceptions	NR	1 duplicate per tank or as required by DQOs Criteria: ±3σ	1 set of MS and MSD per tank or as required by DQO criteria: ±2σ	DQR	GC/MS-tune Surrogate std. Internal std.	1/12 h Each calibration std., each blank, Each sample MS and MSD Each calibration std. each blank Each sample MS and MSD
Radionuclides--Pu, Am, Cs, Np, total alpha	AP1N or whenever check std is out of control	Background 1/24 h[b] AEA background 1/wk Criteria: ≤±3σ	Check std 1/24 h Criteria: ±3σ	1/batch Criteria: ≤±3σ	1/batch Criteria: ±3σ	2 sets of duplicates per tank or as required by DQOs Criteria: RPD ≤±3σ	Each sample ^{236}Pu ^{243}Am ^{242}Pu ^{239}Np or spike Criteria: >30% or ±3σ	Specified for normal sample size, count time, background, and detector each result should include a 2σ counting error	Plateau Beta sensitivity	1/12 mo 1 per month

Table D-6. Quality Control Guidelines for Characterization of Single-Shell Tank Wastes. (sheet 5 of 6)

Parameter	Calibration	Calibration blank[a]	Calibration verification	Preparation blank[a]	Laboratory control standard	Duplicates	Matrix spikes	Detection limits	Other	Requirement
Radionuclides-- ^{90}Sr, ^{99}Tc, ^{63}Ni, ^{129}I, ^{79}Se, total beta	APIN or whenever check standard is out of control & efficiency for each isotope analyzed	Background 1/24 h Criteria: ≤3σ	Check std 1/24 h Criteria: ±3σ	1/batch Criteria: ≤3σ	1/batch Criteria: ±3σ	2 sets of duplicates per tank or as required by DQOs Criteria: RPD ≤3σ	Each sample ^{85}Sr, ^{95}Tc, ^{85}Kr carrier, ^{99}Tc carrier, ^{129}I, carrier, ^{79}Se, carrier Criteria: >50% or ±3σ	Specified for normal sample size count time background and detector each result should include a 2σ counting error	Plateau Beta purity	1/12 mo See discussion
Radionuclides-- ^{14}C, H_3, ^{99}Tc, ^{79}Se, ^{129}I, ^{63}Ni, liquid scintillation	APIN or whenever check standard is out of control	1 background each batch of samples Criteria: ≤3σ	1 check std each batch of samples Criteria: ±3σ	1/batch Criteria: ≤3σ	1/batch Criteria: ±3σ	2 sets of duplicates per tank or as required by DQOs Criteria: RPD ≤3σ	1 spike per core or with matrix change or carrier on each sample Criteria: >50% or ±3σ	Specified for normal sample size, count time, background is system each result should include a 2σ counting error	Quench curve Beta purity	APIN See discussion
Radionuclides-- GEA ^{137}Cs, ^{129}I	APIN or whenever check std is out of control but at least 1/yr	Background 1/24 h Criteria: ≤3σ	Check std 1/24 h Criteria: ±3σ GEA energy adjustment 1/24 h	1/batch Criteria: ≤3σ	1/batch Criteria: ±3σ	2 sets of duplicates per tank or as required by DQOs Criteria: RPD ≤3σ	^{129}I carrier for each sample GEA--NA Criteria >50% or ±3σ	Specified for normal sample size, count time, background, detector for each GEA isotope in library each positive result should include a 2σ counting error	Resolution	1/mo
Radionuclides-- U, Pu-isotopics	APIN	NA	Each batch	Each batch	Each batch	2 sets of duplicates per tank or as required by DQOs	NA	Specified for normal sample size, separation, and instrument	NA	NA
Physical--bulk density	NA	NA	NA	NA	1/batch	2 sets of duplicates per tank if aliquots used or as required by DQOs	NA	NA	NA	NA
Particle size	APIN	NA	1/batch	NA	1/batch	2 sets of duplicates per tank or as required by DQOs	NA	NA	NA	NA
Penetrometer	APIN	NA	NA	NA	NA	2 readings per segment	NA	NA	NA	NA
Physical										

Table D-6. Quality Control Guidelines for Characterization of Single-Shell Tank Wastes. (sheet 6 of 6)

Parameter	Calibration	Calibration blank[a]	Calibration verification	Preparation blank[a]	Laboratory control standard	Duplicates	Matrix spikes	Detection limits	Other	Requirement
Weight percent H_2O	Balance 1/3 mo	NA	Internal--each batch	NA	1/batch	Each sample	NA	NA	NA	NA
Ignitability--reactivity (DSC)	APIN or 1/quarter	APIN	1/batch	NA	1/batch	2 sets of duplicates per tank or for each sample with an exotherm	NA	NA	NA	NA
TCLP	Same as metals	Same as metals	Same as metals	1/batch	Same as metals	1 set of duplicates per tank	1 post extraction spike for each metal for 1 TCLP extraction or with matrix change	Same as metals	NA	NA
Viscosity	APIN	NA	1 per batch or APIN	NA	NA	2 sets of duplicates per tank if sufficient sample available	NA	NA	NA	NA
Shear stress shear rate rheogram and yield stress	APIN	NA	1/batch or APIN	NA	1/batch	2 sets of duplicates per tank if sufficient sample available	NA	NA	NA	NA
Shear strength	APIN no stds.	NA	APIN no stds.	NA	No stds.	2 sets of duplicates per tank if sufficient sample available	NA	NA	NA	NA

[a]The historical mean and 3 σ limit shall be used to evaluate blank method performance; however, other factors such as concentration threshold limit and analyte concentration in sample shall be considered in any corrective action.

[b]Criteria for duplicate and matrix results the same as for ICP.

[c]Calibration blank, preparation blank, and laboratory control standard criteria the same as ICP.

[d]Cool time 0 to 1 h, 1 to 8 h, 8 h to 24 h, 1/24 h, 1/wk.

[e]If ^{99}Tc is not available a ^{99}Tc spike that is 5 to 10 times greater than ^{99}Tc in sample should be used for one sample from each core.

APIN = as per instrument manufacturer.
CC = core composite.
CCC = calibration check compounds.
CRDL = contract required detection limits.
DOE = Define and document detection limit and reestablish any time instrument or procedure changes occur that may impact method performance.
MS = matrix spike.
MSA = method of standard additions.
MSD = matrix spike duplicate.
NA = not applicable.
NR = not required.
RPD = relative percent difference.
RRF = relative response factor.
SPCC = system performance check compounds.
TBD = to be determined or method not developed.
IDL = instrument detection limits.

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11.0 LABORATORY PERFORMANCE AND SYSTEM AUDITS

Audits shall be preceded by a specific sequence of steps that identify the appropriate performance criteria. This in turn establishes the basis for the audit requirements. The items that verify these steps shall be in place prior to the performance of any Environmental Quality Assurance (EQA) audit. These items are as follows:

1. The statement of work (SOW)--has been agreed to by the laboratory and by the customer who has need for the analysis information.

2. The procedures to be used in the performance of work by the laboratory--have been submitted, reviewed, and accepted by the customer for compliance with the SOW.

Each laboratory performing work for the single shell tanks project shall have its own internal guidance for performance and system audits which shall be in compliance with the SOW. Westinghouse Hanford Laboratories (WHC) and Battelle Laboratories (PNL) have developed quality assurance and control for this oversight as described in their respective quality assurance project plans. Audits shall be performed by Environmental Quality Assurance (EQA) to verify the laboratory quality programs are effective.

Environmental Quality Assurance auditing of WHC, PNL, or subcontract laboratories will follow two basic formats and will be applied uniformly to everyone. The only significant difference between the requirements for internal and external audits is in the authority, which is defined by WHC policy guidance for internal audits and within the procurement contract for external audits.

Systems audits as defined in QAMS 005/80 part 5.12 shall be the continuing means of demonstrating compliance of systems to the requirements of the SOW. These audits shall be performed in accordance with *WHC-CM-4-2, QI 10.4, SURVEILLANCE* to evaluate performance of individual systems. The systems audit shall be performed initially when a system specified in the SOW becomes operational and then on a random basis in accordance with a schedule prepared and maintained by EQA after receipt of the approved SOW.

A performance audit as defined in QAMS 005/80 part 5.12 shall be the basis to determine the compliance of the laboratory to the SOW. This audit shall be performed after systems are operational and generating data and biannually there after. The audit shall be controlled in accordance with *WHC-CM-4-8, QAI 18.1, PLANNING, PERFORMING, REPORTING, FOLLOW-UP, AND CLOSURE OF QUALITY ASSURANCE AUDITS*. Credit may be taken for other audit qualification of the laboratory if it establishes reasonable confidence in the performance of analysis for this project. Defense Waste Technology shall be required to review and accept the audit results before the start of work.

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12.0 PREVENTIVE MAINTENANCE

Preventive maintenance policies for the laboratories are described in their respective QAPjPs (WHC 1989, PNL 1989).

The sampling operation and each laboratory shall have a preventive maintenance program to ensure sampling and analysis equipment is kept in proper working order. Maintenance logs shall be maintained for each major piece of equipment to track equipment problems and down times. Adequate spare parts for major equipment shall be maintained to prevent excessive down times for normal repairs.

Since most analytical equipment is based on electronic rather than mechanical systems, preventive maintenance is minimal or not normally required for most systems. Instrument problems are normally identified with the daily control requirements of setting up the equipment and analyzing samples. Both Westinghouse Hanford and PNL have access to in-house instrument repair groups who are capable of troubleshooting and handling many routine repairs.

Calibration and instrument performance shall be checked before resuming sample analysis following any maintenance activity that may affect the data.

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13.0 DATA ASSESSMENT PROCEDURES

The data assessment procedures for the laboratories are described in their respective QAPjPs (WHC 1989, PNL 1989). Accuracy for the SST program shall be assessed by tracking and collecting percent recovery data (for all Type I and Type II analytes) on laboratory control standards. The calibration verification results shall be assessed (tabulated or plotted) by the analyst or chemist in charge of the analysis. The mean recovery and relative standard deviation of this data shall be determined, tracked by tabulating or control charts with 2σ warning limits and 3σ out-of-control limits, and updated on a regular frequency. Method performance (accuracy) on the sample matrix shall be assessed in the same manner using matrix spikes.

The precision of the data results shall be assessed by the relative percentage differences (RPD) of duplicate analyses or matrix spike duplicates for all Type I and Type II analytes. If the duplicate sample analytes results are less than five times the method detection limit, they are subject to large differences because of procedure limitations. This data should be analyzed separately and used to evaluate method precision near the detection limit. The other data would represent the variability at or above a reasonable quantitation level. The data shall be collected, tracked by tabulation or control charts, and updated regularly (after analysis of each batch of tanks). If one of the duplicate results is a less than value, the data is reported but the data are not used to calculate the RPD and is not plotted on the control charts. The use of matrix spike duplicates ensures that duplicate data above detection limits are obtained.

Control charts or tabulated data shall be used to track the background levels of analytes that generate positive values most frequently for sampling, hot cell, calibration, and preparation blanks. Radiochemical instrument backgrounds shall be recorded, plotted, and analyzed for each detector. For single parameter detectors the mean and standard deviation shall be determined and used to set up control limits for identifying changing and out-of-control conditions. For multiple isotope detectors, isotopes that most frequently contribute to the background shall be tracked such as ^{137}Cs for GEA systems and $^{239/240}Pu$ for AEA systems. Control charts (or tabulations) and statistical evaluations shall be performed on other quality control information that may be specific to a technique such as CRDL standards for ICP and GFAA and SPCC and CCC standards for organic analysis.

Control charts (or tabulations) and statistical limits shall be established to assess the reproducibility of homogenization and compositing procedures. This requires the determination of the RPD for the duplicates and monitoring the mean and standard deviation of the data to evaluate the performance of these procedures on different waste types.

Standard statistical methods shall be used to establish the mean and 3σ limits for SST waste characterization data.

Data will be evaluated and validated by the Office of Sample Management using the criteria and data flags described in the procedure "Data Validation for RCRA Analyses" found in the Sample Management Administrative Manual, WHC-CM-5-3.

14.0 CORRECTIVE ACTION

The corrective action procedures are defined in the respective laboratory QAPjPs (WHC 1989, PNL 1989). Limits for data acceptability will be established based on DQOs and laboratory performance. These criteria will be specified in this document as DQOs are developed and as the laboratory performance database is established. These criteria shall be evaluated on a continuous basis during the characterization program to ensure that the laboratory capabilities meet the DQOs. Criteria for each measurement parameter have been defined in Section 10 and Table D-6. Corrective actions have been described in the text of this section. Fundamentally the following guidelines shall be used in correcting SST characterization data.

- Systems found to be out of calibration shall be shut down and recalibrated before rerunning the samples analyzed in the batch prior to the problem and before running any other samples. An exception to this guide may be permissible for multielement techniques in which only one element is out-of-control and it is a low priority analyte. Any exception must be authorized by the Technical Leader, the data flagged, and documented including an estimate of the bias caused by the system.

- Duplicate RPD results for type I and II analytes that exceed limits shall be evaluated with respect to nearness to detection limit and sample heterogeneity. If the sample concentration is greater than 5 times the method detection limit, the duplicate should be rerun (providing enough sample is available) to determine if the problem is an analytical error or sample heterogeneity. Data from the rerun should be reported with each batch and an explanation provided. If the analyte concentration is less than 5 times the detection limit no action is required.

- Spike results for type I and II analytes that exceed limits shall be evaluated with respect to the relative concentration of the spike concentration to the sample concentration and sample heterogeneity. If the duplicate data indicates that the sample is homogeneous a post digestion spike (when applicable) shall be used to evaluate instrument interferences. If no duplicate data is available a duplicate sample shall be run to evaluate sample heterogeneity. If the ratio of spike to sample is too small the spiked sample shall be rerun with a larger spike if practical or another technique such as serial dilution will be used to evaluate the potential interference. All evaluation data shall be included with the batch and explanations for the results documented.

- High blank results which significantly impact an analysis shall require identifying the source of the high blank and removing it when practical. If sample results are impacted by greater than 20% because of the high blank they shall be rerun with a lower blank. If data with lower blank values cannot be obtained the data shall be identified in the results.

- Other measurement systems, procedures or plan corrections that may be required as a result of routine review processes shall be resolved as required by governing procedures or shall be referred to the Technical Lead for resolution. Copies of all surveillance, audit and corrective action documentation shall be considered project QA records upon completion or closure.

15.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

The QA reporting procedures for the laboratories are described in their respective QAPjPs (WHC 1989, PNL 1989). In addition, EQA will provide reports to management as described in the QAPP (WHC 1990).

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GLOSSARY

Accuracy	The closeness of agreement between an observed value and an accepted reference value. When applied to a set of observed values, accuracy will be a combination of a random component and of a common systematic error (or bias) component.
Batch	A group of samples which behave similarly with respect to the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will be handled as a separate batch. For SSTs all the segments taken from a tank plus the core composites prepared from the segments represent a "tank batch" (i.e. for each batch one sampling equipment rinse blank, one hot cell blank, one spike per core, and one duplicate per core will be prepared). For a set of samples digested, fused or prepared as a unit a "preparation batch" will require a method (preparation) blank, a laboratory control standard and may include a spiked or duplicate sample along with the samples prepared at that time. For a set of samples analyzed as a unit an "analysis batch" will require a reagent or calibration blank, calibration verification standard plus any samples, spikes, duplicates, standards and blanks generated in the other operations.
Calibration Verification	Analysis of an independent (different from calibration) standard prior to analysis of samples and at a set frequency to check instrument calibration and performance.
Calibration Blank	Analysis of reagents used in preparation of the calibration which are not processed through the preparation procedure. For example: The acid-water mixture used in ICP dilutions.
Carrier	A non-radioactive spike of known concentration added in a radiochemical separation to aid in separating (precipitating) the isotope of interest and used to correct for incomplete recoveries of the isotope in the separation.
Concentration Threshold Limit	The point at which an analyte begins to make a significant contribution to a risk or waste classification calculation. For the current analysis a "significant contribution" is the level of analyte that provides at least 1% of the total risk index or classification calculation.

Data Quality Objective (DQOs)	Statements of the level of uncertainty that a decision maker is willing to accept in results derived from environmental data. This is qualitatively distinct from quality measurements such as precision, bias, and detection limits.
Duplicate	Two subsamples or aliquots taken from the same segment or core composite sample, that are analyzed to document the precision of a method in a given sample matrix.
Field Blank	A blank sample prepared in the field to evaluate potential contamination from the sampling location, sample equipment or transportation operation.
Hot Cell Blank	A blank sample prepared in the hot cell to evaluate potential contamination from the hot cell environment, extrusion equipment and sample storage containers.
Instrument Detection Limit (IDL)	The instrument detection limit is the point where the measured value is larger than the uncertainty associated with it. This point is defined as 3 times the standard deviation of the measurement as the concentration approaches zero ($3S_o$). For an instrument the S_o may be based on the standard deviation of the blank or the background noise level. The CLP program for ICP and AA determines the IDL by multiplying by 3 the average standard deviation for 7 consecutive measurements on a standard containing analytes at 3 to 5 times the instrument manufacturers suggested IDL for 3 non-consecutive days.
Completeness	A measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct normal conditions.
Check Standard	A check standard is a radiochemical standard that is not prepared with each batch of analyses but is a standard used over an extended period of time to monitor (check) the performance of a radiochemical detector and measurement system on a daily basis.
Control Limits	A range within which specified measurements must fall to be compliant. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that non compliant data be flagged. For SW-846 (RCRA) measurements control limits are normally set at 3 times the standard deviation of the measurement parameter.

Laboratory Control Standard	A laboratory control standard (LCS) is a standard that is carried through the entire preparation and analysis steps of the procedure. The standard may be a liquid, similar but independent from a calibration standard, or a liquid or solid standard containing matrix components. For radiochemical methods where isotope availability is limited, the LCS may be carried only through the separation and measurement procedures.
Long Term Release Risk (LTRR)	Long term release risk concerns are based on health risks to the public over a time period greater than a single life-time (70 yr) to determine potential health effects to current and future generations. The LTRR scenario considers release, transport and toxicity parameters in computing potential risk indexes that can be used to rank analytes according to their potential health risks to the public.
Matrix Detection Limit	This is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is grater than zero and is determined from analysis of a sample in a given matrix containing the analyte. RCRA (SW-846) procedures define this as the method detection limit and is determined by multiplying by 7 the standard deviation obtained from triplicate analyses of a matrix spike containing the analyte of interest at a concentration 3 to 5 times the estimated method detection limit. The estimated method detection limit is on (1) an instrument signal to noise ratio within the range of 2.5 to 5.0 or (2) the region of the standard curve where there is a significant change in sensitivity; i.e., a break in the slope of the standard curve.
Matrix Spike	An aliquot of sample spiked with a known concentration of the analyte(s) being measured. For chemical parameters where the efficiency of the digestion is being evaluated the spike is added prior to digestion. For radiochemical analytes spiking prior to digestion may not be practical because of the availability of spiking isotopes. In which case the matrix spike is added prior to the isotope separation procedure to evaluate the efficiency of the separation and analytical procedures. A matrix spike is used to document the bias of a method in a given sample matrix.

Matrix Spike Duplicate	Intralaboratory split samples spiked with identical concentrations of the analyte(s) being measured. They are used to document the precision and bias of a method in a given sample matrix.
Method Detection Limit	This is the minimum concentration that can be detected in a sample after it has undergone all routine sample preparation and analysis steps. It is determined by multiplying the instrument detection limit by all the normal dilution factors. The method detection limits are reported as μg or μCi per gram of sample. The method detection limit does not account for any changes in sensitivity or background caused by the sample matrix.
Method of Standard Additions	The addition of multiple (3) increments of a standard solution (spikes) to sample aliquots of the same size. Measurements are made on the original and after each addition. The slope, x-intercept and y-intercept are determined by least-square analysis. The analyte concentration is determined by the absolute value of the x-intercept. Spike volume is maintained low (< 10%) of the sample volume. Standard additions may counteract matrix effects; however, it will not counteract spectral effects.
Post Digestion Spike	A spike added after a sample has been digested or leached to evaluate method performance in which the subsampling and digestion/leach procedure reproducibility is not a factor in spike recovery. Normally used for TCLP and water leaches. Also used to help differentiate between sample heterogeneity and instrument or analysis matrix problems.
Preparation Blank	The preparation blank is a test utilizing no sample, or deionized water that is carried through the entire measurement system (i.e., sample digestion, analyte separation and final measurement procedure). It is used to assess contamination levels in the analytical process. This blank is often referred to as a method blank in RCRA procedures.
Sample Breakdown	The operation of loading SST samples in the hot cell, extruding, homogenizing and compositing the samples and preparing subsamples for subsequent analyses.

Short Term Intruder Risk (STIR)	STIR is the risk associated from the possibility of individuals coming into contact with the toxic or radioactive constituent that may pose a threat to personal health. The U.S. Nuclear Regulatory Commission (NRC) outlined three generic intruder scenarios. The Intruder-Construction Scenario, the Intruder-Discovery Scenario and the Intruder-Agriculture Scenario. Exposure routes for these scenarios are ingestion, inhalation and radiological ground exposure.
Surrogate	An organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.
Tracer	A tracer is a radiochemical spike which is a different isotope of the same element that is added to a sample in a known quantity to determine the efficiency or yield of the analytical separation and analysis. Since the tracer isotope has different radiological properties than the isotope of interest the spike recovery and isotope analysis are performed on the same sample aliquot. A "matrix spike" of the same isotope being analyzed will require two sample aliquots.
Traveler	Documentation that accompanies a sample that is used to: (1) provide information to the analysts for performing a procedure such as sample number, analyte and procedure, sample size etc., and (2) document time, date, analyst name, results and observations made during performance of the procedure.
Waste Classification (CLASS)	Waste classification refers to a system of ranking analytes based on regulatory guidelines for disposal of radioactive and chemically hazardous waste. The system is based on the Washington State Department of Ecology method for determining toxic equivalent concentrations and on the U.S. Nuclear Regulatory Commission low-level radioactive waste classification.

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